

# SEARCH REQUEST FORM

Scientific and Technical Information Center

Requester's Full Name: Susy Tsang-Foster Examiner #: 76063 Date: 4/30/03  
 Art Unit: 1745 Phone Number 301-50588 Serial Number: 09/918464  
 Mail Box and Bldg/Room Location: CP38A09 Results Format Preferred (circle) PAPER DISK E-MAIL

If more than one search is submitted, please prioritize searches in order of need.

\*\*\*\*\*

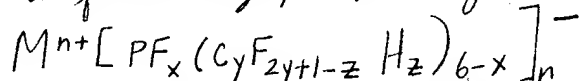
Please provide a detailed statement of the search topic, and describe as specifically as possible the subject matter to be searched. Include the elected species or structures, keywords, synonyms, acronyms, and registry numbers, and combine with the concept or utility of the invention. Define any terms that may have a special meaning. Give examples or relevant citations, authors, etc, if known. Please attach a copy of the cover sheet, pertinent claims, and abstract.

Title of Invention: Fluoroalkyl phosphates for use in electrochemical cells  
 Inventors (please provide full names): Heider et al. (please see attached)

Earliest Priority Filing Date: 8/4/2000

\*For Sequence Searches Only\* Please include all pertinent information (parent, child, divisional, or issued patent numbers) along with the appropriate serial number.

Please search for a fluoroalkyl phosphate of the formula (I)



where  $1 \leq x \leq 6$ ;  $1 \leq y \leq 8$ ;  $0 \leq z \leq 2y+1$ ;  $1 \leq n \leq 3$  and

$M^{n+}$  is a monovalent to trivalent cation where

$M^{n+}$  is NOT Li<sup>+</sup>, Na<sup>+</sup>, Cs<sup>+</sup>, K<sup>+</sup>, and Ag<sup>+</sup>

If there are too many hits, please limit  $M^{n+}$  to be selected from: (please see claim 2) these are excluded. please see claim 1

$NR^1R^2R^3R^4$ ,  $PR^1R^2R^3R^4$ ,  $P(NR^1R^2)_k R^3_m R^4_{4-k-m}$

(where  $k=1-4$ ,  $m=0-3$  and  $k+m \leq 4$ ),

$C(NR^1R^2)(NR^3R^4)(NR^5R^6)$ ,  $C(aryl)_3$ ,  $Rb$ , or tropylium where  $R^1$  to  $R^6$  are H, alkyl, or aryl (substituted by F, Cl, Br).

## STAFF USE ONLY

Searcher: <u>John Calve</u>	Type of Search <u>ST</u>	Vendors and cost where applicable
Searcher Phone #: _____	NA Sequence (#) <u>2</u>	STN _____
Searcher Location: _____	AA Sequence (#) _____	Dialog _____
Date Searcher Picked Up: <u>5/2/03</u>	Structure (#) <u>1</u>	Questel/Orbit _____
Date Completed: <u>5/2/03</u>	Bibliographic _____	Dr. Link _____
Searcher Prep & Review Time: <u>240 min</u>	Litigation _____	Lexis/Nexis _____
Clerical Prep Time: _____	Fulltext _____	Sequence Systems _____
Online Time: <u>120 min</u>	Patent Family _____	WWW/Internet _____
	Other _____	Other (specify) _____

BEST AVAILABLE COPY

Susy,

My structure search is for the P attached to at least one fluorinated alkyl and at least one fluoride. At that point I took out the structures with Li, Na, .... There were 10 records in Chemical Abstracts so I printed them all. The first 3 records have good dates as references.

The second set of answers from the structure search are the the fluorinate phosphorous without the counter ion (M). I printed them just for your information.

John

=> file reg

FILE 'REGISTRY' ENTERED AT 14:26:25 ON 02 MAY 2003  
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PLEASE SEE "HELP USAGETERMS" FOR DETAILS.  
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Property values tagged with IC are from the ZIC/VINITI data file provided by InfoChem.

STRUCTURE FILE UPDATES: 30 APR 2003 HIGHEST RN 508165-25-1  
DICTIONARY FILE UPDATES: 30 APR 2003 HIGHEST RN 508165-25-1

TSCA INFORMATION NOW CURRENT THROUGH JANUARY 6, 2003

=> d his

(FILE 'HOME' ENTERED AT 14:02:38 ON 02 MAY 2003)

FILE 'LREGISTRY' ENTERED AT 14:02:52 ON 02 MAY 2003

L1 STR  
L2 SCR 2040

FILE 'REGISTRY' ENTERED AT 14:13:13 ON 02 MAY 2003

L3 4 S L1 AND L2  
L4 98 S L1 AND L2 FULL  
SAVE L4 FOSTER464/A  
L5 26 S L4 AND 1-10/N

FILE 'LREGISTRY' ENTERED AT 14:15:21 ON 02 MAY 2003

FILE 'REGISTRY' ENTERED AT 14:16:26 ON 02 MAY 2003

L6 62 S L4 AND 2-10/NC  
L7 49 S L6 NOT 1-2/LI  
L8 48 S L7 NOT 1-2/NA  
L9 43 S L8 NOT 1-3/CS  
L10 38 S L9 NOT 1-3/K  
L11 36 S L10 NOT 1-3/AG

FILE 'HCA' ENTERED AT 14:19:54 ON 02 MAY 2003

L12 10 S L11

L13 FILE 'CAOLD' ENTERED AT 14:20:32 ON 02 MAY 2003  
0 S L11

L14 FILE 'HCA' ENTERED AT 14:21:23 ON 02 MAY 2003  
629 S HEIDER ?/AU  
L15 47856 S SCHMIDT ?/AU  
L16 100 S KUHNER ?/AU  
L17 1503 S SARTORI ?/AU  
L18 81 S IGNATYEV ?/AU  
L19 4 S L14 AND L15 AND L16  
L20 10 S L12 NOT L19  
E US20020022182/PN  
L21 1 S E3  
L22 9 S L12 NOT L21  
L23 180233 S BATTERY? OR BATTERIES? OR (ELECTROCHEM? OR ELECTRO(W)CHEM? OR  
L24 5 S L20 AND L23  
L25 3 S (L20 OR L24) AND 1950-2000/PY  
L26 7 S L20 NOT L25  
L27 7 S L26 OR L24  
L28 36 S L4 NOT L6

L29 FILE 'HCA' ENTERED AT 14:31:09 ON 02 MAY 2003  
25 S L28  
L30 22 S L29 AND 1930-2000/PY  
L31 0 S L30 AND L23

L32 FILE 'REGISTRY' ENTERED AT 14:32:55 ON 02 MAY 2003  
7 S L28 AND (C(L)H(L)P(L)M)/ELS  
L33 16 S L28 NOT 1-20/O

L34 FILE 'HCA' ENTERED AT 14:38:51 ON 02 MAY 2003  
14 S L33  
L35 13 S L34 AND 1907-2000/PY  
L36 13 S L35 NOT L20

FILE 'REGISTRY' ENTERED AT 14:26:25 ON 02 MAY 2003

=> d que stat L4

L1 STR

7  
F  
>  
C~P~Ak~F  
1 2 3 4

NODE ATTRIBUTES:  
DEFAULT MLEVEL IS ATOM  
DEFAULT ECLEVEL IS LIMITED

GRAPH ATTRIBUTES:  
RING(S) ARE ISOLATED OR EMBEDDED  
NUMBER OF NODES IS 5

STEREO ATTRIBUTES: NONE  
L2 SCR 2040  
L4 98 SEA FILE=REGISTRY SSS FUL L1 AND L2

100.0% PROCESSED 15392 ITERATIONS  
SEARCH TIME: 00.00.01

98 ANSWERS

=&gt; file hca

FILE 'HCA' ENTERED AT 14:26:36 ON 02 MAY 2003

USE IS SUBJECT TO THE TERMS OF YOUR STN CUSTOMER AGREEMENT.

PLEASE SEE "HELP USAGETERMS" FOR DETAILS.

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FILE COVERS 1907 - 1 May 2003 VOL 138 ISS 19

FILE LAST UPDATED: 1 May 2003 (20030501/ED)

This file contains CAS Registry Numbers for easy and accurate substance identification.

=&gt; d L25 1-3 ibib abs hitstr hitrn

L25 ANSWER 1 OF 3 HCA COPYRIGHT 2003 ACS

ACCESSION NUMBER: 111:174247 HCA

TITLE: Reaction of tris(perfluoroalkyl)phosphine oxides and tris(perfluoroalkyl)difluorophosphoranes with fluoride ion

AUTHOR(S): Pavlenko, N. V.; Yagupol'skii, L. M.

CORPORATE SOURCE: Inst. Org. Khim., Kiev, USSR

SOURCE: Zhurnal Obshchei Khimii (1989), 59(3), 528-34

CODEN: ZOKHA4; ISSN: 0044-460X

DOCUMENT TYPE: Journal

LANGUAGE: Russian

OTHER SOURCE(S): CASREACT 111:174247

AB Treating (C2F5)3P(O) with 1 or 2 equiv. CsF in Et2O gave (C2F5)3PFOCs or (C2F5)2PF2OCs, resp.; hydrolysis of the latter gave C2F5P(O)FOCs. Treating R3PF2 (R = C2F5, C3F7, C4F9) with MF (M = Cs, K, Na) in Et2O gave quant. M+[R3PF3]-. Diazotization of 4-XC6H4NH2 (X = Cl, Me, NO2) and subsequent ~~reaction~~ with K+[R3PF3]- (R = C2F5, C3F7) gave 77-88% [4-XC6H4N2]+[R3PF3]-.

IT 123199-70-2P 123199-72-4P 123199-73-5P

123199-74-6P

RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)  
(prepn. and thermal decompn. of)

RN 123199-70-2 HCA

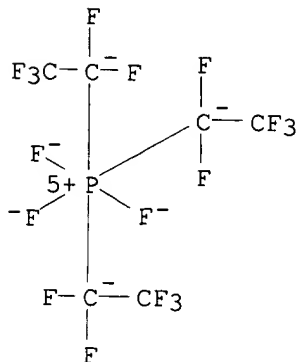
CN Benzenediazonium, 4-chloro-, (OC-6-21)-trifluorotris(pentafluoroethyl)phosphate(1-) (9CI) (CA INDEX NAME)

CM 1

CRN 123199-69-9

CMF C6 F18 P

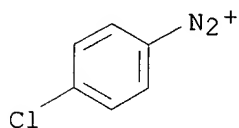
CCI CCS



CM 2

CRN 17333-85-6

CMF C6 H4 Cl N2



RN 123199-72-4 HCA

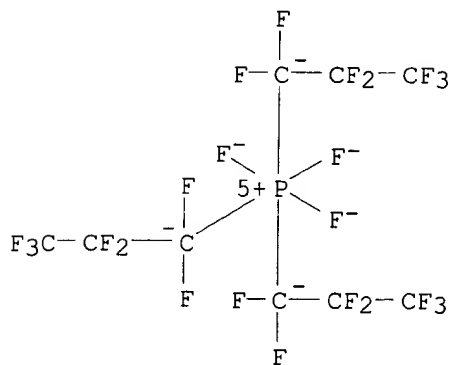
CN Benzenediazonium, 4-chloro-, (OC-6-21)-trifluorotris(heptafluoropropyl)phosphate(1-) (9CI) (CA INDEX NAME)

CM 1

CRN 123199-71-3

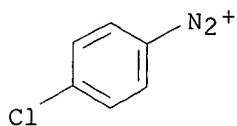
CMF C9 F24 P

CCI CCS



CM 2

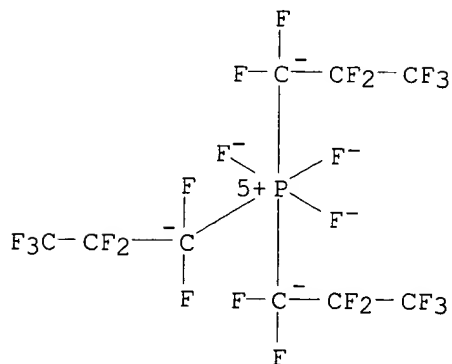
CRN 17333-85-6  
CMF C6 H4 Cl N2



RN 123199-73-5 HCA  
CN Benzenediazonium, 4-methyl-, (OC-6-21)-trifluorotris(heptafluoropropyl)phosphate(1-) (9CI) (CA INDEX NAME)

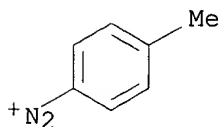
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CRN 123199-71-3  
CMF C9 F24 P  
CCI CCS



CM 2

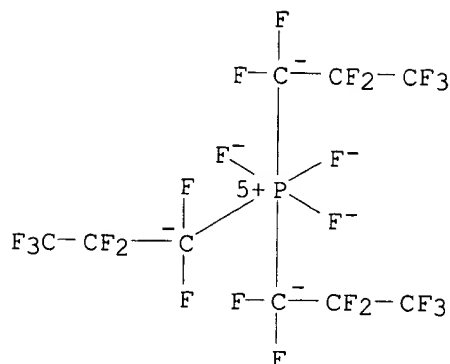
CRN 57573-52-1  
CMF C7 H7 N2



RN 123199-74-6 HCA  
CN Benzenediazonium, 4-nitro-, (OC-6-21)-trifluorotris(heptafluoropropyl)phosphate(1-) (9CI) (CA INDEX NAME)

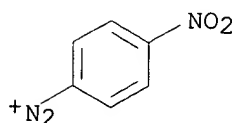
CM 1

CRN 123199-71-3  
CMF C9 F24 P  
CCI CCS



CM 2

CRN 14368-49-1  
CMF C6 H4 N3 O2



IT 123199-70-2P 123199-72-4P 123199-73-5P  
123199-74-6P

RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT  
(Reactant or reagent)  
(prepn. and thermal decompn. of)

L25 ANSWER 2 OF 3 HCA COPYRIGHT 2003 ACS

ACCESSION NUMBER: 71:81483 HCA

TITLE: Formation of trifluoromethylated fluoro phosphates by  
reaction of trimethyltrifluoromethyltin with  
phosphorus(V) fluoride

AUTHOR(S): Jander, Jochen; Boerner, Dieter; Engelhardt, Udo

CORPORATE SOURCE: Freie Univ., Berlin, Fed. Rep. Ger.

SOURCE: Justus Liebigs Annalen der Chemie (1969),  
726, 19-24

CODEN: JLACBF; ISSN: 0075-4617

DOCUMENT TYPE: Journal

LANGUAGE: German

AB PF5 reacted with Me3SnCF3 to give a white hygroscopic ppt. that slowly  
gave off PF5; the anions formed are pptd. from CH2Cl2 as stable  
(Ph4As)(PF5CF3) and (Ph4As)[PF4(CF3)2]. Their structures were established  
from ir and 19F N.M.R. data and a mechanism of formation is discussed.

IT 23940-75-2P

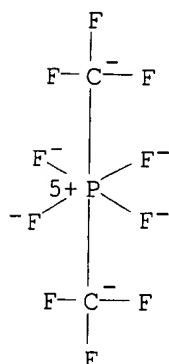
RL: SPN (Synthetic preparation); PREP (Preparation)  
(prepn. of)

RN 23940-75-2 HCA

CN Arsonium, tetraphenyl-, tetrafluorobis(trifluoromethyl)phosphate(1-) (8CI)  
(CA INDEX NAME)

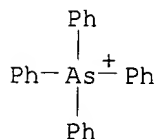
CM 1

CRN 45043-58-1  
CMF C2 F10 P  
CCI CCS



CM 2

CRN 15912-80-8  
CMF C24 H20 As



IT **23940-75-2P**

RL: SPN (Synthetic preparation); PREP (Preparation)  
(prepn. of)

L25 ANSWER 3 OF 3 HCA COPYRIGHT 2003 ACS

ACCESSION NUMBER: 68:114717 HCA

TITLE: Trifluoromethyl-substituted fluorophosphates and fluoroarsenates

AUTHOR(S): Chan, S. S.; Willis Christopher J.

CORPORATE SOURCE: Univ. Western Ontario, London, ON, Can.

SOURCE: Canadian Journal of Chemistry (1968), 46(8),  
1237-48

CODEN: CJCHAG; ISSN: 0008-4042

DOCUMENT TYPE: Journal

LANGUAGE: English

AB Synthetic routes were developed to salts contg. the anions  $[\text{CF}_3\text{PF}_5]^-$ ,  $[(\text{CF}_3)_2\text{PF}_4]^-$ ,  $[(\text{CF}_3)_3\text{AsF}_4]^-$ ,  $[(\text{CF}_3)_2\text{AsF}_4]^-$ , and  $[(\text{CF}_3)_3\text{AsF}_3]^-$ . These are isolated as stable solids with  $\text{Cs}^+$ , or sometimes  $\text{Ag}^+$ , as the cation. Their 19F N.M.R. spectra are discussed, and it is suggested that the bis- and tris(trifluoromethyl)-substituted fluorophosphates have a trans configuration. Trimethyltrifluoromethyltin,  $\text{Me}_3\text{SnCF}_3$ , forms 1:1 complexes with  $\text{PF}_5$ ,  $(\text{CF}_3)_2\text{PF}_3$ , and  $(\text{CF}_3)_3\text{PF}_2$ . It is suggested that transfer of a trifluoromethyl group as  $\text{CF}_3^-$  has occurred here, leading to the formation of trimethyltin derivs. of the trifluoromethyl-substituted fluorophosphates. 22 references.

IT **18757-46-5P 18757-47-6P**

RL: SPN (Synthetic preparation); PREP (Preparation)  
(prepn. of)



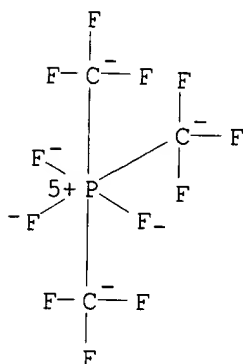
RN 18757-46-5 HCA  
 CN Stannylum, trimethyl-, (OC-6-21)-trifluorotris(trifluoromethyl)phosphate(1-)  
 (9CI) (CA INDEX NAME)

CM 1

CRN 45166-84-5

CMF C3 F12 P

CCI CCS

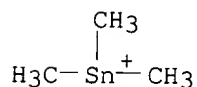


CM 2

CRN 5089-96-3

CMF C3 H9 Sn

CCI CCS



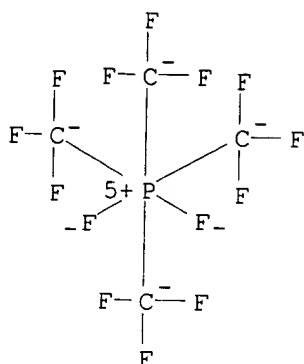
RN 18757-47-6 HCA  
 CN Stannylum, trimethyl-, difluorotetrakis(trifluoromethyl)phosphate(1-)  
 (9CI) (CA INDEX NAME)

CM 1

CRN 45224-05-3

CMF C4 F14 P

CCI CCS

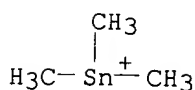


CM 2

CRN 5089-96-3

CMF C3 H9 Sn

CCI CCS



IT 18757-46-5P 18757-47-6P

RL: SPN (Synthetic preparation); PREP (Preparation)  
(prepn. of)

=&gt; d L27 1-7 ibib abs hitind hitstr hitrn

L27 ANSWER 1 OF 7 HCA COPYRIGHT 2003 ACS (Authors Record)  
 ACCESSION NUMBER: 138:92290 HCA  
 TITLE: Synthesis, properties, and uses of  
 (perfluoroalkyl)phosphorane-based novel strong acids  
 and acid salts as catalysts, solvents, ionic liquids,  
 and **battery** electrolytes  
 INVENTOR(S): Ignatyev, Nikolai; Schmidt, Michael; Kuehner, Andreas;  
 Hilarius, Volker; Heider, Udo; Kucheryna, Andriy;  
 Sartori, Peter; Willner, Helge  
 PATENT ASSIGNEE(S): Merck Patent G.m.b.H., Germany  
 SOURCE: PCT Int. Appl., 46 pp.  
 CODEN: PIXXD2  
 DOCUMENT TYPE: Patent  
 LANGUAGE: German  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2003002579	A1	20030109	WO 2002-EP6360	20020611
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, OM, PH, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TN, TR, TT, TZ,				

UA, UG, US, UZ, VN, YU, ZA, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU,  
TJ, TM

RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AT, BE, CH,  
CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR,  
BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG

DE 10130940

A1

20030116

DE 2001-10130940 20010627

PRIORITY APPLN. INFO.:

DE 2001-10130940 A 20010627

OTHER SOURCE(S):

MARPAT 138:92290

AB Novel strong (perfluoroalkyl)phosphorane-type acids and acid salts are of general formulas [RyPF6-y]-.H<sup>+</sup> (I) and [RyPF6-y]m-.Mm<sup>+</sup> (II), in which R = partially fluorinated or perfluoro-C1-8-alkyl or aryl (in which F or H can be substituted by Cl); y = 1-3; m = 1-3, and Mm<sup>+</sup> is a mono-, di-, or trivalent cation (e.g., Li<sup>+</sup>, Zn<sup>2+</sup>, Mg<sup>2+</sup>, Cu<sup>2+</sup>, Ag<sup>+</sup>, ammonium, phosphonium, oxonium, sulfonium, arsonium, tropylium, nitryl, nitrosyl, or tris(dialkylamino)carbonium cations). I are prepd. by reaction of HF with the corresponding (perfluoroalkyl)fluorophosphoranes, RyPF5-y, in the presence of a solvent or a proton acceptor; similarly, II are prepd. from the corresponding I by reaction with a salt, of formula Mm<sup>+</sup>(A)m<sup>-</sup>, in which Mm<sup>+</sup> is as defined above and (A)m<sup>-</sup> is a basic or neutral anion that can react with a proton (e.g., a metal, a metal hydride, a metal oxide, or a metal hydroxide). The acids and salts have use as strong acid catalysts, phase transfer catalysts, solvents, ionic liqs., or conducting salts in electrochem. devices (esp. **battery** electrolytes).

IC ICM C07F009-28

CC 49-8 (Industrial Inorganic Chemicals)

Section cross-reference(s): 52

ST perfluoroalkyl fluorophosphorane strong acid; fluorophosphorane metal salt synthesis; strong acid catalyst **battery** electrolyte

perfluoroalkyl fluorophosphorane

IT Phosphonium compounds

Sulfonium compounds

RL: CAT (Catalyst use); NUU (Other use, unclassified); PRP (Properties);

SPN (Synthetic preparation); PREP (Preparation); USES (Uses)

((perfluoroalkyl)fluorophosphorane salts; synthesis, properties, and uses of (perfluoroalkyl)phosphorane-based novel strong acids and acid salts as catalysts, solvents, ionic liqs., and **battery** electrolytes)

IT Onium compounds

RL: CAT (Catalyst use); NUU (Other use, unclassified); PRP (Properties);

SPN (Synthetic preparation); PREP (Preparation); USES (Uses)

(arsonium, (perfluoroalkyl)fluorophosphorane salts; synthesis, properties, and uses of (perfluoroalkyl)phosphorane-based novel strong acids and acid salts as catalysts, solvents, ionic liqs., and **battery** electrolytes)

IT Onium compounds

RL: CAT (Catalyst use); NUU (Other use, unclassified); PRP (Properties);

SPN (Synthetic preparation); PREP (Preparation); USES (Uses)

(oxonium, (perfluoroalkyl)fluorophosphorane salts; synthesis, properties, and uses of (perfluoroalkyl)phosphorane-based novel strong acids and acid salts as catalysts, solvents, ionic liqs., and **battery** electrolytes)

IT Amines, uses

RL: NUU (Other use, unclassified); USES (Uses)

(polyamines, nonpolymeric, solvent or proton acceptors; synthesis, properties, and uses of (perfluoroalkyl)phosphorane-based novel strong acids and acid salts as catalysts, solvents, ionic liqs., and **battery** electrolytes)

IT Alcohols, uses

Amines, uses

Carboxylic acids, uses

- Esters, uses  
Ethers, uses  
Glycols, uses  
Polysulfides  
Sulfides, uses  
RL: NUU (Other use, unclassified); USES (Uses)  
(solvent or proton acceptors; synthesis, properties, and uses of (perfluoroalkyl)phosphorane-based novel strong acids and acid salts as catalysts, solvents, ionic liqs., and **battery** electrolytes)
- IT Acids, preparation  
RL: CAT (Catalyst use); NUU (Other use, unclassified); PRP (Properties); SPN (Synthetic preparation); PREP (Preparation); USES (Uses)  
(strong; synthesis, properties, and uses of (perfluoroalkyl)phosphorane-based novel strong acids and acid salts as catalysts, solvents, ionic liqs., and **battery** electrolytes)
- IT **Battery** electrolytes  
Catalysts  
Ionic liquids  
Phase transfer catalysts  
(synthesis, properties, and uses of (perfluoroalkyl)phosphorane-based novel strong acids and acid salts as catalysts, solvents, ionic liqs., and **battery** electrolytes)
- IT Phosphoranes  
RL: CAT (Catalyst use); NUU (Other use, unclassified); PRP (Properties); SPN (Synthetic preparation); PREP (Preparation); USES (Uses)  
(synthesis, properties, and uses of (perfluoroalkyl)phosphorane-based novel strong acids and acid salts as catalysts, solvents, ionic liqs., and **battery** electrolytes)
- IT 22474-72-2  
RL: RCT (Reactant); RACT (Reactant or reagent)  
(reaction of, with HF; synthesis, properties, and uses of (perfluoroalkyl)phosphorane-based novel strong acids and acid salts as catalysts, solvents, ionic liqs., and **battery** electrolytes)
- IT 7664-39-3, Hydrogen fluoride, reactions  
RL: RCT (Reactant); RACT (Reactant or reagent)  
(reactions of, with phosphoranes; synthesis, properties, and uses of (perfluoroalkyl)phosphorane-based novel strong acids and acid salts as catalysts, solvents, ionic liqs., and **battery** electrolytes)
- IT 60-29-7, Diethyl ether, uses 64-17-5, Ethanol, uses 64-19-7, Acetic acid, uses 67-56-1, Methanol, uses 68-12-2, Dimethyl formamide, uses 75-18-3, Dimethyl sulfide 115-10-6, Dimethyl ether 121-44-8, Triethylamine, uses 603-35-0, Triphenylphosphine, uses 616-38-6, Dimethyl carbonate 7732-18-5, Water, uses 7803-51-2, Phosphine  
RL: NUU (Other use, unclassified); USES (Uses)  
(solvent or proton acceptors; synthesis, properties, and uses of (perfluoroalkyl)phosphorane-based novel strong acids and acid salts as catalysts, solvents, ionic liqs., and **battery** electrolytes)
- IT 7439-93-2DP, Lithium, (perfluoroalkyl)fluorophosphorane salts  
7439-95-4DP, Magnesium, (perfluoroalkyl)fluorophosphorane salts  
7440-22-4DP, Silver, (perfluoroalkyl)fluorophosphorane salts  
7440-50-8DP, Copper, (perfluoroalkyl)fluorophosphorane salts  
7440-66-6DP, Zinc, (perfluoroalkyl)fluorophosphorane salts 14452-93-8DP, Nitrosyl, (perfluoroalkyl)fluorophosphorane salts 14522-82-8DP, Nitryl, (perfluoroalkyl)fluorophosphorane salts 14798-03-9DP, Ammonium, (perfluoroalkyl)fluorophosphorane salts 25215-10-5DP, Guanidine, conjugate monoacid, alkyl derivs., (perfluoroalkyl)fluorophosphorane salts 26811-28-9DP, Tropylium, (perfluoroalkyl)fluorophosphorane salts  
**482635-70-1P 482635-71-2P 482635-72-3P**  
**482635-73-4P 482649-24-1P**, Trifluorotris(heptafluoro-1-propyl)phosphate, acid salt **482649-25-2P**,

Trifluorotris(nonafluoro-1-butyl)phosphate, acid salt  
 RL: CAT (Catalyst use); NUU (Other use, unclassified); PRP (Properties);  
 SPN (Synthetic preparation); PREP (Preparation); USES (Uses)  
 (synthesis, properties, and uses of (perfluoroalkyl)phosphorane-based  
 novel strong acids and acid salts as catalysts, solvents, ionic liqs.,  
 and **battery** electrolytes)

IT 403699-22-9P **463944-41-4P** 482635-76-7P **482635-77-8P**  
**482635-78-9P** **482635-79-0P** 482635-80-3P  
**482635-81-4P** **482635-83-6P**

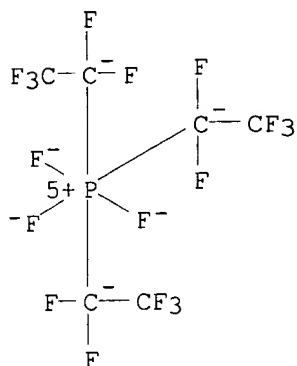
RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation)  
 (synthesis, properties, and uses of (perfluoroalkyl)phosphorane-based  
 novel strong acids and acid salts as catalysts, solvents, ionic liqs.,  
 and **battery** electrolytes)

IT **482635-70-1P** **482635-71-2P** **482635-72-3P**  
**482635-73-4P** **482649-24-1P**, Trifluorotris(heptafluoro-1-  
 propyl)phosphate, acid salt **482649-25-2P**,

Trifluorotris(nonafluoro-1-butyl)phosphate, acid salt  
 RL: CAT (Catalyst use); NUU (Other use, unclassified); PRP (Properties);  
 SPN (Synthetic preparation); PREP (Preparation); USES (Uses)  
 (synthesis, properties, and uses of (perfluoroalkyl)phosphorane-based  
 novel strong acids and acid salts as catalysts, solvents, ionic liqs.,  
 and **battery** electrolytes)

RN 482635-70-1 HCA

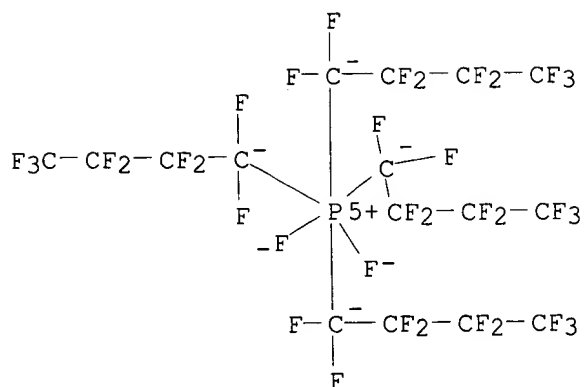
CN Phosphate(1-), trifluorotris(pentafluoroethyl)-, hydrogen (9CI) (CA INDEX  
 NAME)



● H<sup>+</sup>

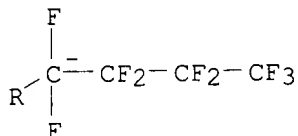
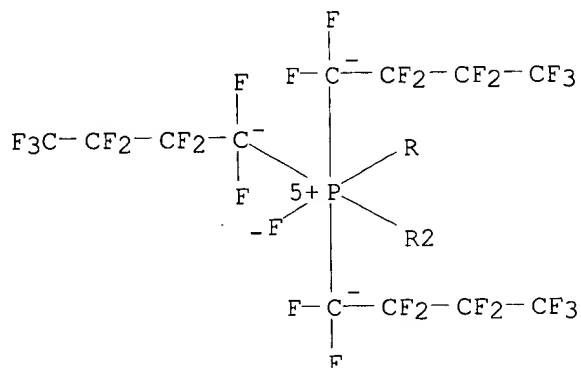
RN 482635-71-2 HCA

CN Phosphate(1-), difluorotetrakis(nonafluorobutyl)-, hydrogen (9CI) (CA  
 INDEX NAME)

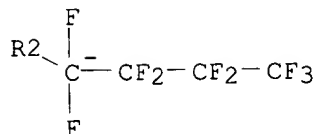


RN 482635-72-3 HCA  
 CN Phosphate(1-), fluoropentakis(nonafluorobutyl)-, hydrogen, (OC-6-21)-  
 (9CI) (CA INDEX NAME)

PAGE 1-A

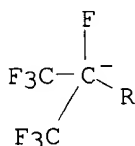
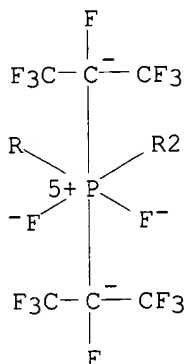


PAGE 2-A

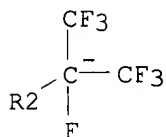


RN 482635-73-4 HCA  
 CN Phosphate(1-), difluorotetrakis[1,2,2,2-tetrafluoro-1-(trifluoromethyl)ethyl]-, hydrogen (9CI) (CA INDEX NAME)

PAGE 1-A

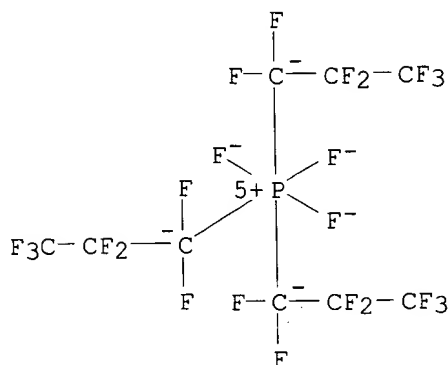


PAGE 2-A



RN 482649-24-1 HCA

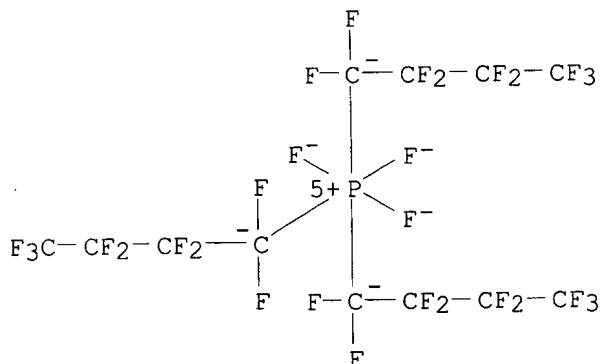
CN Phosphate(1-), trifluorotris(heptafluoropropyl)-, hydrogen (9CI) (CA INDEX NAME)



● H<sup>+</sup>

RN 482649-25-2 HCA

CN Phosphate(1-), trifluorotris(nonafluorobutyl)-, hydrogen (9CI) (CA INDEX NAME)



● H<sup>+</sup>

IT 463944-41-4P 482635-77-8P 482635-78-9P  
482635-79-0P 482635-81-4P 482635-83-6P

RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation)  
(synthesis, properties, and uses of (perfluoroalkyl)phosphorane-based  
novel strong acids and acid salts as catalysts, solvents, ionic liqs.,  
and **battery** electrolytes)

RN 463944-41-4 HCA

CN Ethanaminium, N,N,N-triethyl-, trifluorotris(pentafluoroethyl)phosphate(1-)  
(9CI) (CA INDEX NAME)

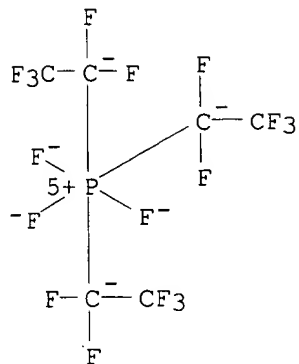
CM 1

CRN 429679-87-8

CMF C6 F18 P

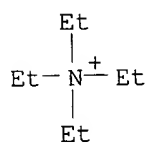
CCI CCS



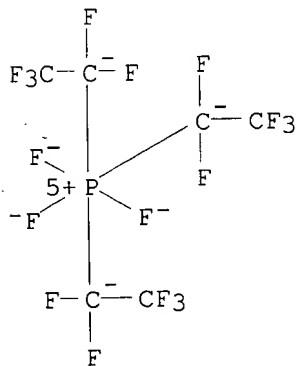


CM 2

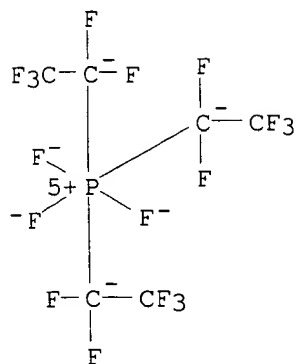
CRN 66-40-0  
CMF C8 H20 N



RN 482635-77-8 HCA  
CN Phosphate(1-), trifluorotris(pentafluoroethyl)-, magnesium (2:1) (9CI)  
(CA INDEX NAME)

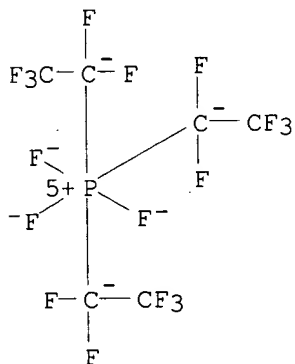
● 1/2 Mg<sup>2+</sup>

RN 482635-78-9 HCA  
CN Phosphate(1-), trifluorotris(pentafluoroethyl)-, zinc (2:1) (9CI) (CA  
INDEX NAME)



● 1/2 Zn<sup>2+</sup>

RN 482635-79-0 HCA  
 CN Phosphate(1-), trifluorotris(pentafluoroethyl)-, copper(2+) (2:1) (9CI)  
 (CA INDEX NAME)

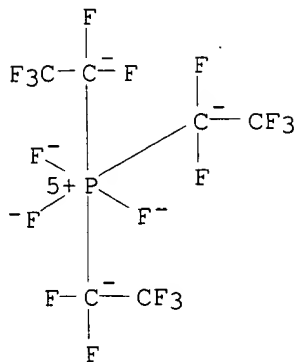


1/2 Cu(II) 2+

RN 482635-81-4 HCA  
 CN Phosphonium, tetrabutyl-, trifluorotris(pentafluoroethyl)phosphate(1-)  
 (9CI) (CA INDEX NAME)

CM 1

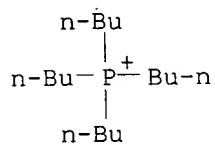
CRN 429679-87-8  
 CMF C6 F18 P  
 CCI CCS



CM 2

CRN 15853-37-9

CMF C16 H36 P



RN 482635-83-6 HCA

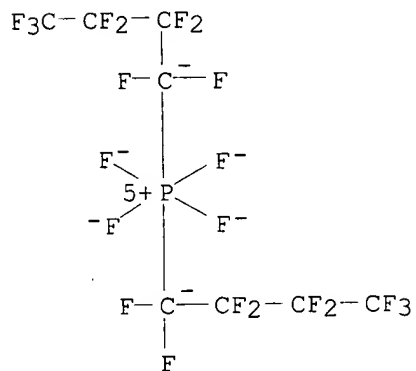
CN Ethanaminium, N,N,N-triethyl-, tetrafluorobis(nonafluorobutyl)phosphate(1-)  
(9CI) (CA INDEX NAME)

CM 1

CRN 482635-82-5

CMF C8 F22 P

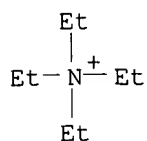
CCI CCS



CM 2

CRN 66-40-0

CMF C8 H20 N



IT 482635-70-1P 482635-71-2P 482635-72-3P  
 482635-73-4P 482649-24-1P, Trifluorotris(heptafluoro-1-propyl)phosphate, acid salt 482649-25-2P,  
 Trifluorotris(nonafluoro-1-butyl)phosphate, acid salt  
 RL: CAT (Catalyst use); NUU (Other use, unclassified); PRP (Properties);  
 SPN (Synthetic preparation); PREP (Preparation); USES (Uses)  
 (synthesis, properties, and uses of (perfluoroalkyl)phosphorane-based  
 novel strong acids and acid salts as catalysts, solvents, ionic liqs.,  
 and **battery** electrolytes)

IT 463944-41-4P 482635-77-8P 482635-78-9P  
 482635-79-0P 482635-81-4P 482635-83-6P  
 RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation)  
 (synthesis, properties, and uses of (perfluoroalkyl)phosphorane-based  
 novel strong acids and acid salts as catalysts, solvents, ionic liqs.,  
 and **battery** electrolytes)

REFERENCE COUNT: 9 THERE ARE 9 CITED REFERENCES AVAILABLE FOR THIS  
 RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L27 ANSWER 2 OF 7 HCA COPYRIGHT 2003 ACS  
 ACCESSION NUMBER: 137:265682 HCA  
 TITLE: Procedure for the production of fluoroalkylphosphates  
 INVENTOR(S): Schmidt, Michael; Kuehner, Andreas; Jungnitz, Michael;  
 Ott, Frank; Ignatyev, Nicolai  
 PATENT ASSIGNEE(S): Merck Patent G.m.b.H., Germany  
 SOURCE: Ger., 8 pp.  
 CODEN: GWXXAW  
 DOCUMENT TYPE: Patent  
 LANGUAGE: German  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 10119278	C1	20021002	DE 2001-10119278	20010420
JP 2002356491	A2	20021213	JP 2001-297523	20010927
WO 2002085919	A1	20021031	WO 2002-EP3288	20020323

W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN,  
 CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH,  
 GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR,  
 LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, OM, PH,  
 PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TN, TR, TT, TZ,  
 UA, UG, US, UZ, VN, YU, ZA, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU,  
 TJ, TM

RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AT, BE, CH,  
 CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR,  
 BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG

PRIORITY APPLN. INFO.: DE 2001-10119278 A 20010420

OTHER SOURCE(S): MARPAT 137:265682

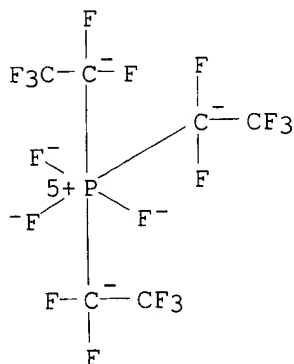
AB A procedure to produce F alkyl phosphates with the general formula  
 $\text{Mn}^+[\text{PF}_x(\text{CyF}_2\text{y}+1-\text{zHz})_{6-x}]_n$  where 1.ltoreq.x.ltoreq.6, 1.ltoreq.y.ltoreq.8,  
 0.ltoreq.z.ltoreq.2y+1, 1.ltoreq.n.ltoreq.3 and Mn<sup>+</sup> is a mono-, di- or  
 trivalent cation. Suitable cations include Li; Na; K; Mg; Rb; Cs; arom.  
 heterocyclic cations; NR<sub>1</sub>R<sub>2</sub>R<sub>3</sub>R<sub>4</sub>; PR<sub>1</sub>R<sub>2</sub>R<sub>3</sub>R<sub>4</sub>; P[(NR<sub>1</sub>R<sub>2</sub>)<sub>k</sub>R<sub>3</sub>mR<sub>4</sub>4-k-m] with

$k=1-4$ ,  $m=0,3$  and  $k+m<4$ ;  $C(NR_1R_2)(NR_3R_4)(NR_5R_6)$ ;  $C(Aryl)_3$ ; Rb or Tropylium; and where  $R_1$  to  $R_6$  is H, Alkyl and Aryl(C1-C8) that can be partially substituted with F, Cl or Br. The fluoroalkylphosphoranes are converted into the required product with metal or nonmetal fluorides without using solvents in the process. These salts are suitable electrolytes in **batteries**, condensers, supercondensers and **galvanic cells**.

IC ICM C07F009-52  
 CC 52-2 (Electrochemical, Radiational, and Thermal Energy Technology)  
 Section cross-reference(s): 29, 72  
 ST fluoroalkylphosphate prodn electrolyte **battery** condenser  
**galvanic cell**  
 IT **Battery** electrolytes  
 Solid electrolytes  
 (prodn. of fluoroalkylphosphate electrolytes)  
 IT 205926-54-1P 206057-04-7P **463944-41-4P** 463944-42-5P  
 RL: IMF (Industrial manufacture); PREP (Preparation)  
 (prodn. of fluoroalkylphosphates)  
 IT **463944-41-4P**  
 RL: IMF (Industrial manufacture); PREP (Preparation)  
 (prodn. of fluoroalkylphosphates)  
 RN 463944-41-4 HCA  
 CN Ethanaminium, N,N,N-triethyl-, trifluorotris(pentafluoroethyl)phosphate(1-)  
 ) (9CI) (CA INDEX NAME)

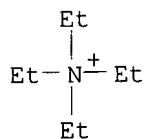
CM 1

CRN 429679-87-8  
 CMF C6 F18 P  
 CCI CCS



CM 2

CRN 66-40-0  
 CMF C8 H20 N



IT **463944-41-4P**

RL: IMF (Industrial manufacture); PREP (Preparation)

(prodn. of fluoroalkylphosphates)

REFERENCE COUNT: 1 THERE ARE 1 CITED REFERENCES AVAILABLE FOR THIS  
RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L27 ANSWER 3 OF 7 HCA COPYRIGHT 2003 ACS

ACCESSION NUMBER: 137:217076 HCA

TITLE: Preparation of fluoroalkylphosphate salts as  
electrolytes for primary and secondary  
**batteries**INVENTOR(S): Schmidt, Michael; Kuehner, Andreas; Ignatyev, Nikolai;  
Satori, Peter

PATENT ASSIGNEE(S): Merck Patent G.m.b.H., Germany

SOURCE: Eur. Pat. Appl., 26 pp.

CODEN: EPXXDW

DOCUMENT TYPE: Patent

LANGUAGE: German

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 1236732	A1	20020904	EP 2002-1914	20020131
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR				
DE 10109032	A1	20020905	DE 2001-10109032	20010224
JP 2003034692	A2	20030207	JP 2001-301156	20010928
CN 1371911	A	20021002	CN 2002-105228	20020221
BR 2002000465	A	20021029	BR 2002-465	20020221
US 2002122979	A1	20020905	US 2002-80515	20020225

PRIORITY APPLN. INFO.: DE 2001-10109032 A 20010224

OTHER SOURCE(S): CASREACT 137:217076; MARPAT 137:217076

AB The prepn. of title compds., useful as electrolytes for primary and  
secondary **batteries**, is described. Thus, reaction of LiF with  
perfluoro-1,2-bis(diethyldifluorophosphorano)ethane in a mixt. of ethylene  
carbonate/dimethyl carbonate/diethyl carbonate (solvent mixt.) gave the  
title compd., 2Li+[(C2F5)2PF3(CF2)2PF3(C2F5)]2-, as a mixt. of  
stereoisomers. The oxidn. stability of the compd. prepd. is given.

IC ICM C07F009-28

CC 29-7 (Organometallic and Organometalloidal Compounds)

Section cross-reference(s): 52, 72

IT Electrolytes

(prepn. of fluoroalkylphosphate salts as electrolytes for primary and  
secondary **batteries**)IT Secondary **batteries**(primary and; prepn. of fluoroalkylphosphate salts as electrolytes for  
primary and secondary **batteries**)

IT Oxidation

(stability; prepn. of fluoroalkylphosphate salts as electrolytes for  
primary and secondary **batteries**)

IT 454458-13-0P

RL: CPS (Chemical process); PEP (Physical, engineering or chemical  
process); PRP (Properties); RCT (Reactant); SPN (Synthetic preparation);  
PREP (Preparation); PROC (Process); RACT (Reactant or reagent)(oxidn. stability; prepn. of fluoroalkylphosphate salts as electrolytes  
for primary and secondary **batteries**)

IT 403699-22-9P 454458-15-2P

RL: SPN (Synthetic preparation); PREP (Preparation)

(prepn. of fluoroalkylphosphate salts as electrolytes for primary and  
secondary **batteries**)

IT 60-29-7, Diethyl ether, uses 67-64-1, Acetone, uses 67-68-5, DMSO, uses 68-12-2, DMF, uses 75-05-8, Acetonitrile, uses 75-18-3, Dimethyl sulfide 79-20-9, Methyl acetate 96-48-0, .gamma.-Butyrolactone 96-49-1, Ethylene carbonate 105-37-3, Ethyl propionate 105-54-4, Ethyl butyrate 105-58-8, Diethyl carbonate 107-13-1, Acrylonitrile, uses 107-31-3, Methyl formate 108-32-7, Propylene carbonate 109-94-4, Ethyl formate 110-71-4 127-19-5, Dimethylacetamide 141-78-6, Ethyl acetate, uses 352-93-2, Diethyl sulfide 554-12-1, Methyl propionate 598-03-8 616-38-6, Dimethyl carbonate 623-42-7, Methyl butyrate 623-53-0, Ethyl methyl carbonate 4437-85-8, Butylene carbonate 56525-42-9, Methyl propyl carbonate 73506-93-1, Diethoxyethane  
 RL: NUU (Other use, unclassified); USES (Uses)  
 (solvent; prepn. of fluoroalkylphosphate salts as electrolytes for primary and secondary **batteries**)

IT **454458-15-2P**  
 RL: SPN (Synthetic preparation); PREP (Preparation)  
 (prepn. of fluoroalkylphosphate salts as electrolytes for primary and secondary **batteries**)

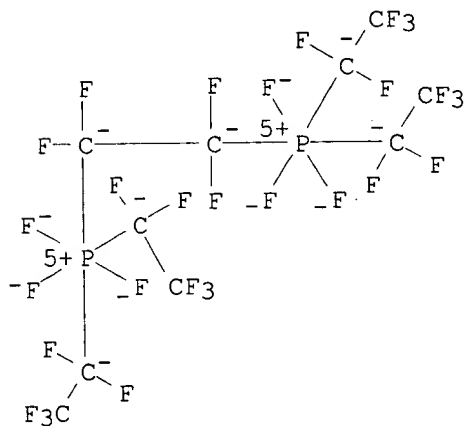
RN 454458-15-2 HCA  
 CN Ethanaminium, N,N,N-triethyl-, hexafluorotetrakis(pentafluoroethyl)[.mu.-(1,1,2,2-tetrafluoro-1,2-ethanediyl)]diphosphate(2-) (2:1) (9CI) (CA INDEX NAME)

CM 1

CRN 454458-14-1

CMF C10 F30 P2

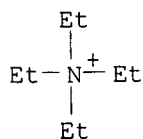
CCI CCS



CM 2

CRN 66-40-0

CMF C8 H20 N



IT 454458-15-2P

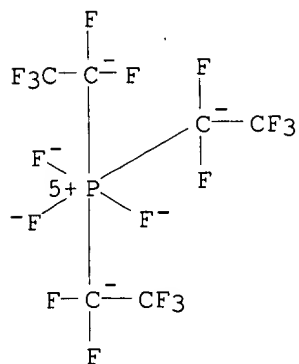
RL: SPN (Synthetic preparation); PREP (Preparation)  
(prepn. of fluoroalkylphosphate salts as electrolytes for primary and  
secondary **batteries**)

REFERENCE COUNT: 2 THERE ARE 2 CITED REFERENCES AVAILABLE FOR THIS  
RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L27 ANSWER 4 OF 7 HCA COPYRIGHT 2003 ACS  
ACCESSION NUMBER: 136:410180 HCA  
TITLE: Nonaqueous electrolytes for electrochemical capacitors  
INVENTOR(S): Takeda, Masayuki; Takehara, Masahiro; Ue, Makoto  
PATENT ASSIGNEE(S): Mitsubishi Chemical Corp., Japan  
SOURCE: Jpn. Kokai Tokkyo Koho, 7 pp.  
CODEN: JKXXAF  
DOCUMENT TYPE: Patent  
LANGUAGE: Japanese  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
-----	---	-----	-----	-----
JP 2002151361	A2	20020524	JP 2000-347833	20001115
PRIORITY APPLN. INFO.:			JP 2000-347833	20001115
AB	The nonaq. electrolytes contain quaternary onium salts expressed as the formula: $Q^+[(R_f)_nRF_6-n]$ , where $Q^+$ is onium ion, $R_f$ is perfluoroalkyl group, $n$ is integer 1-6. When $n$ is .gtoreq.2, plural no. of $R_f$ s can be same or different, and they can bond to form ring structure along with P. The electrolytes are not likely to hydrolytically decompd. compared to tetrafluoroborate.			
IC	ICM H01G009-038 ICS H01G009-00; H01M010-40			
CC	76-10 (Electric Phenomena) Section cross-reference(s): 72			
IT	429679-86-7 <b>429679-88-9 429679-90-3</b> RL: DEV (Device component use); USES (Uses) (nonaq. electrolytes for electrochem. capacitors)			
IT	<b>429679-88-9 429679-90-3</b> RL: DEV (Device component use); USES (Uses) (nonaq. electrolytes for electrochem. capacitors)			
RN	429679-88-9 HCA			
CN	Ethanaminium, N,N-diethyl-N-methyl-, trifluorotris(pentafluoroethyl)phosph ate(1-) (9CI) (CA INDEX NAME)			
CM	1			
CRN	429679-87-8			
CMF	C6 F18 P			
CCI	CCS			

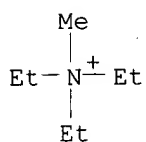




CM 2

CRN 302-57-8

CMF C7 H18 N



RN 429679-90-3 HCA

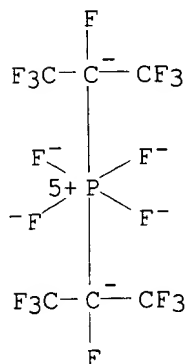
CN Methanaminium, N,N,N-trimethyl-, tetrafluorobis[1,2,2,2-tetrafluoro-1-(trifluoromethyl)ethyl]phosphate(1-) (9CI) (CA INDEX NAME)

CM 1

CRN 429679-89-0

CMF C6 F18 P

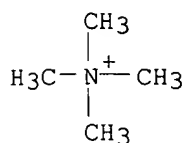
CCI CCS



CM 2 .

CRN 51-92-3

CMF C4 H12 N



IT 429679-88-9 429679-90-3

RL: DEV (Device component use); USES (Uses)  
(nonaq. electrolytes for electrochem. capacitors)

L27 ANSWER 5 OF 7 HCA COPYRIGHT 2003 ACS

ACCESSION NUMBER: 136:388473 HCA

TITLE: Perfluoroalkyl phosphate salt, organic solvent, and polymer mixtures as electrolytes

INVENTOR(S): Schmidt, Michael; Ott, Frank; Jungnitz, Michael; Ignatyev, Nicolai; Kuehner, Andreas

PATENT ASSIGNEE(S): Merck Patent GmbH, Germany

SOURCE: Eur. Pat. Appl., 16 pp.

CODEN: EPXXDW

DOCUMENT TYPE: Patent

LANGUAGE: German

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 1205998	A2	20020515	EP 2001-124178	20011011
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR				
DE 10055812	A1	20020523	DE 2000-10055812	20001110
DE 10058264	A1	20020529	DE 2000-10058264	20001123
CN 1353134	A	20020612	CN 2001-137868	20011109
US 2002114996	A1	20020822	US 2001-986773	20011109
BR 2001005142	A	20020625	BR 2001-5142	20011112
JP 2002249670	A2	20020906	JP 2001-346335	20011112
PRIORITY APPLN. INFO.:			DE 2000-10055812 A	20001110
			DE 2000-10058264 A	20001123

OTHER SOURCE(S): MARPAT 136:388473

AB Electrolytes for **batteries**, condensers, supercondensers, and **galvanic cells** consist of: (1) a fluoroalkyl phosphate salt of general formula  $\text{Mn}^+ ([\text{PF}_x(\text{CyF}_2\text{y}+1-\text{zHz})_6-x])_n$  in which  $\text{Mn}^+$  is a monovalent, divalent, or trivalent cation,  $x = 1-5$ ;  $1 \leq y \leq 8$ ; and  $z = 2y + 1$ ;  $n = 1-3$ ; and the ligands  $\text{CyF}_2\text{y}+1-\text{zHz}$  are the same or different, (2) an org. solvent, selected from org. carbonates, esters, ethers, amides, a sulfur-contg. solvent, and aprotic solvents, and (3) a polymer. The cation ( $\text{Mn}^+$ ) can be a metal ion (e.g.,  $\text{Li}^+$ ,  $\text{Na}^+$ ,  $\text{K}^+$ ,  $\text{Rb}^+$ ,  $\text{Cs}^+$ ,  $\text{Mg}^{2+}$ , or  $\text{Al}^{3+}$ ), preferably  $\text{Li}^+$ , or an org. cation, such as  $\text{NR}_4^+$ ,  $[\text{P}(\text{NR}_2)_k\text{R}_4-k]^+$  ( $k = 0-4$ ),  $[\text{C}(\text{NR}_2)_3]^+$ , or  $[\text{CR}_3]^+$ . The polymer component is selected from homopolymers or copolymers of vinylidenedifluoride, acrylonitrile, Me (meth)acrylate, or THF (preferably polyvinylidene difluoride).

IC ICM H01M010-40

ICS H01B001-12; H01G009-02; C07F009-28

CC 52-2 (Electrochemical, Radiational, and Thermal Energy Technology)

ST **battery** electrolyte fluoroalkyl phosphate salt org carbonate;  
polymer **battery** electrolyte fluoroalkyl phosphateIT **Battery** electrolytes  
(perfluoroalkyl phosphate salt, org. solvent, and polymer mixts. as electrolytes)

IT 60-29-7, Diethyl ether, uses 67-64-1, Acetone, uses 67-68-5, DMSO, uses 68-12-2, Dimethylformamide, uses 75-05-8, Acetonitrile, uses 79-20-9, Methyl acetate 96-48-0, .gamma.-Butyrolactone 96-49-1, Ethylene carbonate 105-37-3, Ethyl propanoate 105-54-4, Ethyl butyrate 105-58-8, Diethyl carbonate 107-13-1, Acrylonitrile, uses 107-31-3, Methyl formate 108-32-7, Propylene carbonate 109-94-4, Ethyl formate 110-71-4 127-19-5, Dimethylacetamide 141-78-6, Ethyl acetate, uses 463-79-6D, Carbonic acid, alkyl esters 554-12-1, Methyl propanoate 616-38-6, Dimethyl carbonate 616-42-2, Dimethyl sulfite 623-42-7, Methyl butyrate 623-53-0, Ethyl methyl carbonate 623-81-4, Diethyl sulfite 1120-71-4, Propanesultone 4437-85-8, Butylene carbonate 24937-79-9, Polyvinylidene difluoride 56525-42-9, Methyl propyl carbonate 73506-93-1, Diethoxyethane 206057-04-7 377739-48-5  
 394692-80-9 394692-84-3 394692-91-2  
 394692-92-3 394692-93-4 394692-94-5  
 425633-73-4 425633-74-5 425633-75-6  
 425633-76-7

RL: TEM (Technical or engineered material use); USES (Uses)  
 (electrolytes contg.; perfluoroalkyl phosphate salt, org. solvent, and polymer mixts. as electrolytes)

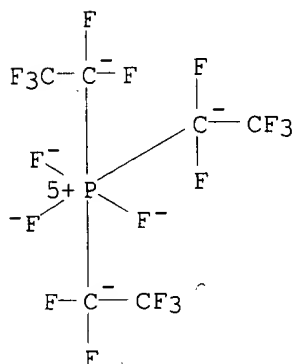
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 425633-76-7

RL: TEM (Technical or engineered material use); USES (Uses)  
 (electrolytes contg.; perfluoroalkyl phosphate salt, org. solvent, and polymer mixts. as electrolytes)

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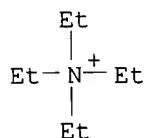
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 CCI CCS



CM 2

CRN 66-40-0  
 CMF C8 H20 N



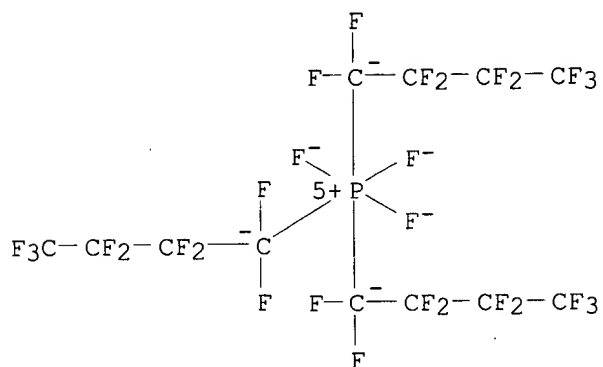
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RN 394692-84-3 HCA
CN Methanaminium, N,N,N-trimethyl-, (OC-6-21)-trifluorotris(nonafluorobutyl)p
   hosphate(1-) (9CI) (CA INDEX NAME)
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CRN 377739-46-3

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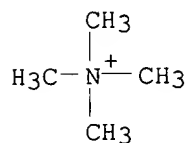
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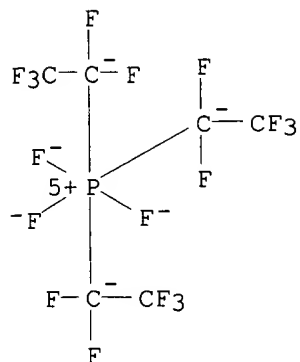
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CRN 123199-69-9

CMF C6 F18 P

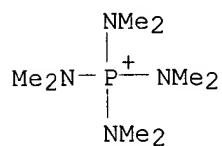
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CRN 45050-74-6

CMF C8 H24 N4 P



RN 394692-92-3 HCA

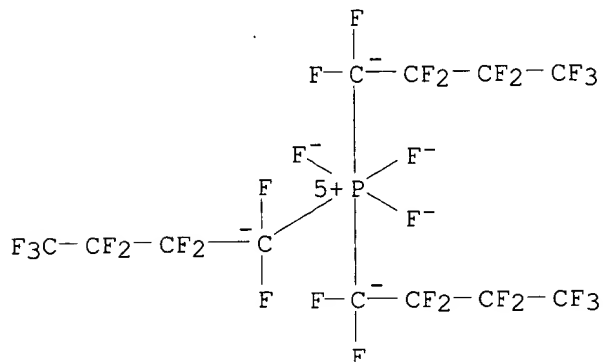
CN Phosphorus(1+), tetrakis(N-methylmethanaminato)-, (T-4)-,  
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 NAME)

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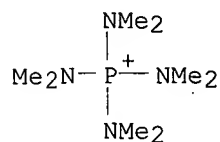
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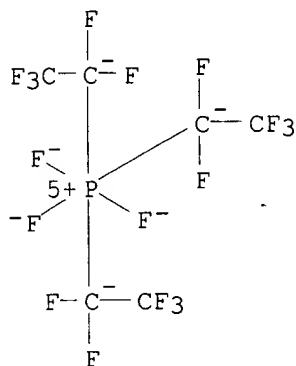
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CRN 123199-69-9

CMF C6 F18 P

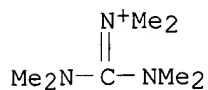
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CM 2

CRN 44872-05-1

CMF C7 H18 N3



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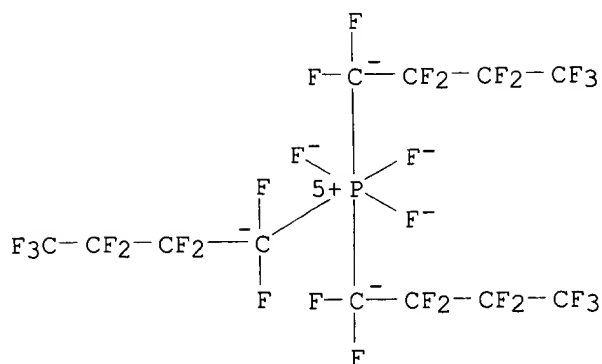
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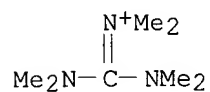
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CRN 44872-05-1

CMF C7 H18 N3



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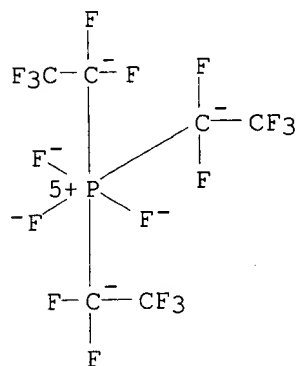
CN Phosphonium, tetramethyl-, (OC-6-21)-trifluorotris(pentafluoroethyl)phosphate(1-) (9CI) (CA INDEX NAME)

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CRN 123199-69-9

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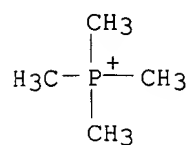
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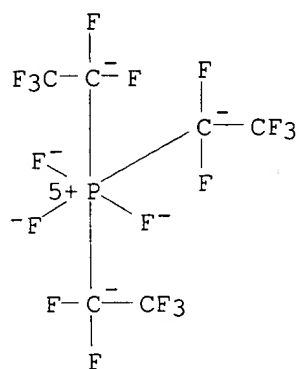
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RN 425633-74-5 HCA  
 CN Phosphonium, tetraethyl-, (OC-6-21)-trifluorotris(pentafluoroethyl)phospha  
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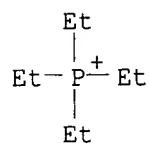
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CRN 13983-95-4  
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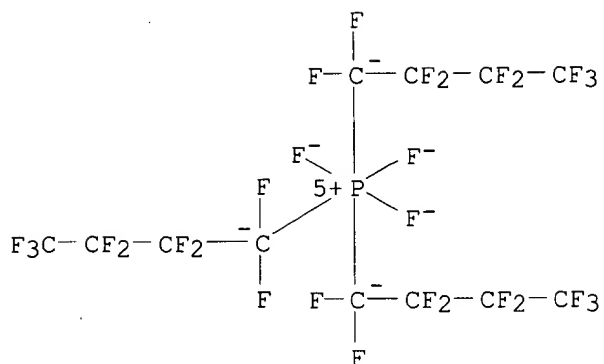


RN 425633-75-6 HCA  
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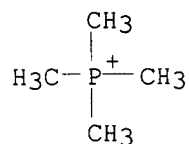




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CMF C4 H12 P



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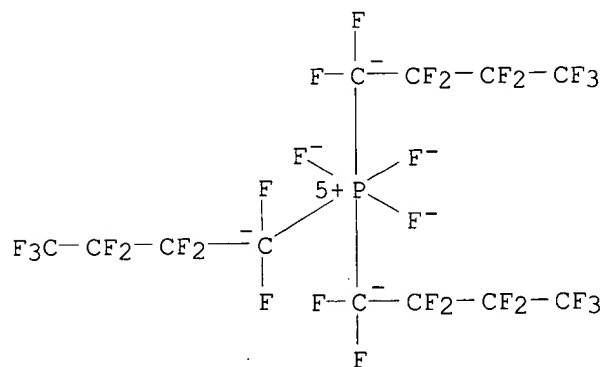
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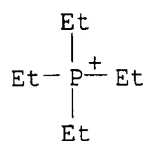
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CM 2

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IT 394692-80-9 394692-84-3 394692-91-2  
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 425633-73-4 425633-74-5 425633-75-6  
 425633-76-7

RL: TEM (Technical or engineered material use); USES (Uses)  
 (electrolytes contg.; perfluoroalkyl phosphate salt, org. solvent, and  
 polymer mixts. as electrolytes)

L27 ANSWER 6 OF 7 HCA COPYRIGHT 2003 ACS

ACCESSION NUMBER: 136:151308 HCA

TITLE: Preparation of fluoroalkylphosphates for use in  
**electrochemical cells**

INVENTOR(S): Heider, Udo; Schmidt, Michael; Kuehner, Andreas;  
 Sartori, Peter; Ignatyev, Nikolai

PATENT ASSIGNEE(S): Merck Patent G.m.b.H., Germany

SOURCE: Eur. Pat. Appl., 15 pp.

CODEN: EPXXDW

DOCUMENT TYPE: Patent

LANGUAGE: German

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 1178050	A2	20020206	EP 2001-115786	20010711
EP 1178050	A3	20020925		
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO				
DE 10038858	A1	20020214	DE 2000-10038858	20000804
US 2002022182	A1	20020221	<u>US 2001-918464</u>	<u>20010801</u>
BR 2001003182	A	20020319	BR 2001-3182	20010802
JP 2002138095	A2	20020514	JP 2001-235045	20010802
CN 1337398	A	20020227	CN 2001-123298	20010803
			DE 2000-10038858 A	20000804

PRIORITY APPLN. INFO.:

OTHER SOURCE(S): MARPAT 136:151308

AB The prepn. of fluoroalkylphosphates via cation exchange reaction is  
 described. Thus, reaction of Li[PF<sub>3</sub>(C<sub>2</sub>F<sub>5</sub>)<sub>3</sub>] with Et<sub>4</sub>NX (X = F, Cl) gave  
 title compds., Et<sub>4</sub>N[PF<sub>3</sub>(C<sub>2</sub>F<sub>5</sub>)<sub>3</sub>]. The prepd. compds. are useful as  
 supporting electrolyte in **batteries**, condensation,  
 supercondensation, and **electrochem. cells**.

IC ICM C07F009-28

ICS C07C211-63; C07C211-14; H01M010-40

CC 29-7 (Organometallic and Organometalloidal Compounds)

Section cross-reference(s): 72

ST fluoroalkyl phosphate prepn **electrolyte electrochem  
 cell**

IT Phosphates, preparation

RL: SPN (Synthetic preparation); TEM (Technical or engineered material  
 use); PREP (Preparation); USES (Uses)

(fluoroalkylphosphates; prepn. of fluoroalkylphosphates for use in  
**electrochem. cells**)

IT **Electrochemical cells**

Electrolytes

## Primary batteries

(prepn. of fluoroalkylphosphates for use in **electrochem.**  
cells)

IT 394692-80-9P 394692-83-2P 394692-84-3P  
394692-91-2P 394692-92-3P 394692-93-4P  
394692-94-5P

RL: SPN (Synthetic preparation); TEM (Technical or engineered material  
use); PREP (Preparation); USES (Uses)

(prepn. of fluoroalkylphosphates for use in **electrochem.**  
cells)

IT 394692-80-9P 394692-83-2P 394692-84-3P  
394692-91-2P 394692-92-3P 394692-93-4P  
394692-94-5P

RL: SPN (Synthetic preparation); TEM (Technical or engineered material  
use); PREP (Preparation); USES (Uses)

(prepn. of fluoroalkylphosphates for use in **electrochem.**  
cells)

RN 394692-80-9 HCA

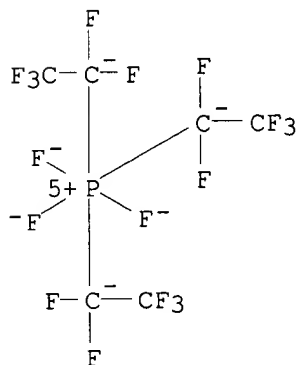
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osphate(1-) (9CI) (CA INDEX NAME)

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CRN 123199-69-9

CMF C6 F18 P

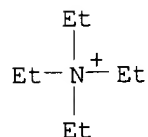
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CM 2

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CMF C8 H20 N

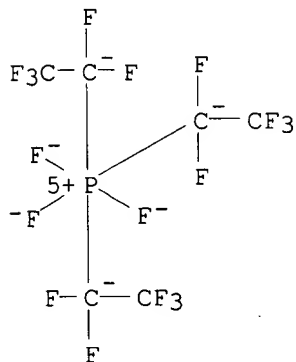


RN 394692-83-2 HCA

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phosphate(1-) (9CI) (CA INDEX NAME)

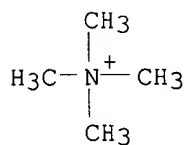
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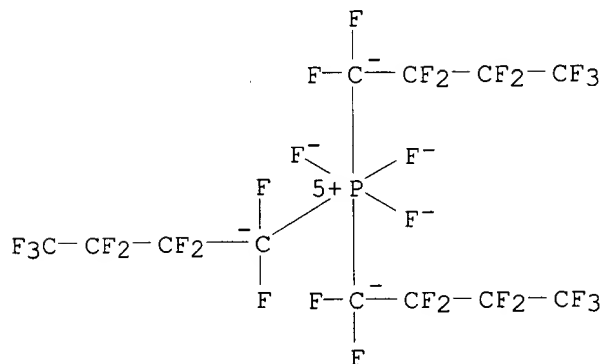
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RN 394692-84-3 HCA  
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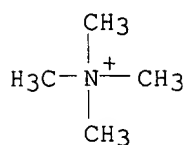
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 CCI CCS



CM 2

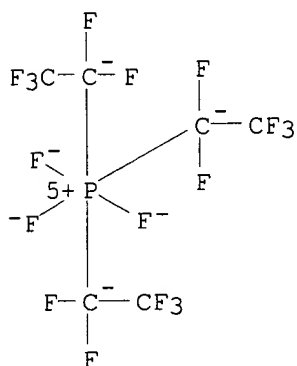
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RN 394692-91-2 HCA  
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(OC-6-21)-trifluorotris(pentafluoroethyl)phosphate(1-) (9CI) (CA INDEX  
NAME)

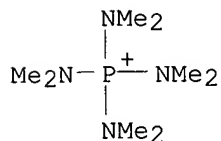
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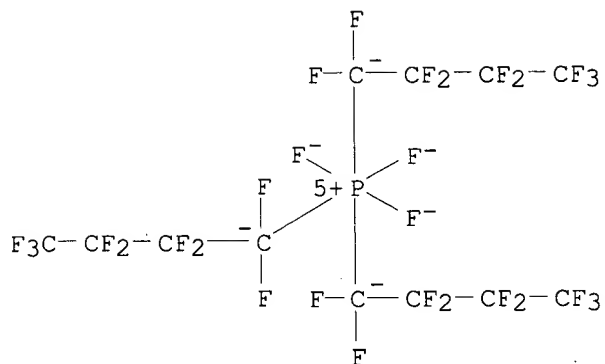
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RN 394692-92-3 HCA  
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NAME)

CM 1

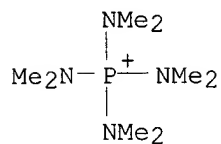
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CMF C8 H24 N4 P



RN 394692-93-4 HCA

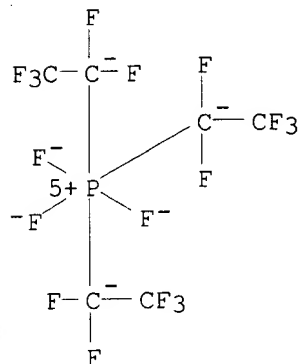
CN Methanaminium, N-[bis(dimethylamino)methylene]-N-methyl-,  
(OC-6-21)-trifluorotris(pentafluoroethyl)phosphate(1-) (9CI) (CA INDEX  
NAME)

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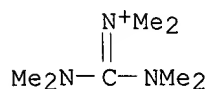
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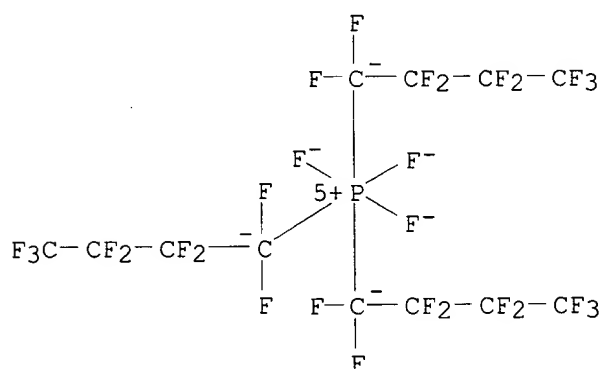
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CRN 44872-05-1

CMF C7 H18 N3

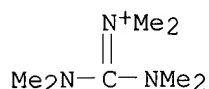


RN 394692-94-5 HCA  
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 (OC-6-21)-trifluorotris(nonafluorobutyl)phosphate(1-) (9CI) (CA INDEX  
 NAME)  
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 CRN 377739-46-3  
 CMF C12 F30 P  
 CCI CCS



CM 2

CRN 44872-05-1  
 CMF C7 H18 N3



IT 394692-80-9P 394692-83-2P 394692-84-3P  
 394692-91-2P 394692-92-3P 394692-93-4P  
 394692-94-5P  
 RL: SPN (Synthetic preparation); TEM (Technical or engineered material  
 use); PREP (Preparation); USES (Uses)  
 (prepn. of fluoroalkylphosphates for use in **electrochem.**  
**cells**)

L27 ANSWER 7 OF 7 HCA COPYRIGHT 2003 ACS  
 ACCESSION NUMBER: 136:20157 HCA  
 TITLE: Ionic liquids  
 INVENTOR(S): Schmidt, Michael; Heider, Udo; Geissler, Winfried;  
 Ignatyev, Nikolai; Hilarius, Volker  
 PATENT ASSIGNEE(S): Merck Patent G.m.b.H., Germany  
 SOURCE: Eur. Pat. Appl., 19 pp.  
 CODEN: EPXXDW  
 DOCUMENT TYPE: Patent

LANGUAGE: German

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 1162204	A1	20011212	EP 2001-111953	20010521
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO				
DE 10027995	A1	20011213	DE 2000-10027995	20000609
CN 1327986	A	20011226	CN 2001-122805	20010608
JP 2002025610	A2	20020125	JP 2001-173307	20010608
BR 2001002318	A	20020213	BR 2001-2318	20010608
US 2002015884	A1	20020207	US 2001-877259	20010611

PRIORITY APPLN. INFO.:

DE 2000-10027995 A 20000609

AB The prepn. of title compds. is described. Thus, reaction of 1-ethyl-3-methylimidazolium chloride with Li[PF<sub>3</sub>(C<sub>2</sub>F<sub>5</sub>)<sub>3</sub>] gave title compd., 1-ethyl-3-methylimidazolium tris(pentafluoroethyl)trifluorophosphate.

IC ICM C07F009-28

ICS H01M010-40; C07D233-58

CC 29-7 (Organometallic and Organometalloidal Compounds)

Section cross-reference(s): 28

IT **377739-43-0P 377739-45-2P 377739-47-4P**

RL: SPN (Synthetic preparation); PREP (Preparation)

(prepn. as ionic liqs.)

IT **377739-43-0P 377739-45-2P 377739-47-4P**

RL: SPN (Synthetic preparation); PREP (Preparation)

(prepn. as ionic liqs.)

RN 377739-43-0 HCA

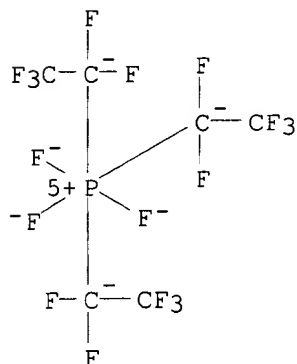
CN 1H-Imidazolium, 1-ethyl-3-methyl-, (OC-6-21)-trifluorotris(pentafluoroethyl)phosphate(1-) (9CI) (CA INDEX NAME)

CM 1

CRN 123199-69-9

CMF C6 F18 P

CCI CCS

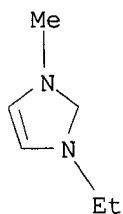


CM 2

CRN 65039-03-4

CMF C6 H11 N2





\*\*\* FRAGMENT DIAGRAM IS INCOMPLETE \*\*\*

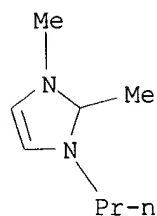
RN 377739-45-2 HCA

CN 1H-Imidazolium, 1,2-dimethyl-3-propyl-, (OC-6-21)-trifluorotris(pentafluoroethyl)phosphate(1-) (9CI) (CA INDEX NAME)

CM 1

CRN 157310-70-8

CMF C8 H15 N2



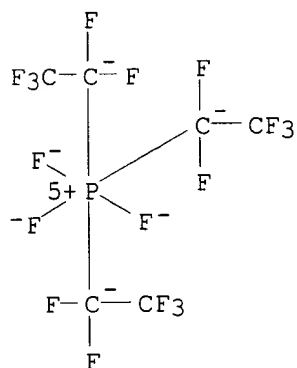
\*\*\* FRAGMENT DIAGRAM IS INCOMPLETE \*\*\*

CM 2

CRN 123199-69-9

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CCI CCS



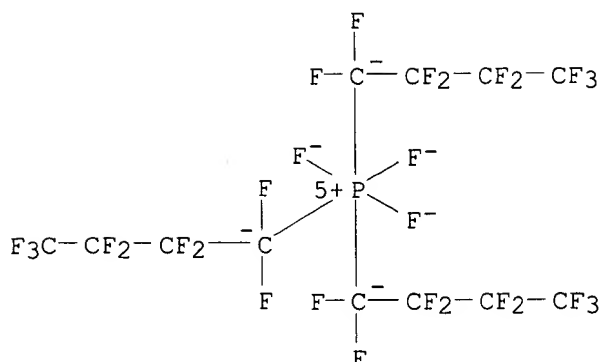
RN 377739-47-4 HCA

CN 1H-Imidazolium, 1-ethyl-3-methyl-, (OC-6-21)-trifluorotris(nonafluorobutyl)phosphate(1-) (9CI) (CA INDEX NAME)

CM 1

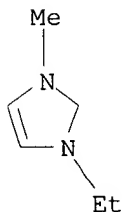
CRN 377739-46-3

CMF C12 F30 P  
CCI CCS



CM 2

CRN 65039-03-4  
CMF C6 H11 N2



\*\*\* FRAGMENT DIAGRAM IS INCOMPLETE \*\*\*

IT 377739-43-0P 377739-45-2P 377739-47-4P

RL: SPN (Synthetic preparation); PREP (Preparation)  
(prepn. as ionic liqs.)

REFERENCE COUNT: 2 THERE ARE 2 CITED REFERENCES AVAILABLE FOR THIS  
RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

\*\*\*\*\*

FYI,

These records don't have the cation (M)).

=> d L36 1,3, 5, 7, 9,11,13 cbib abs fhitstr

L36 ANSWER 1 OF 13 HCA COPYRIGHT 2003 ACS

94:113653 Phosphorus-31 nuclear magnetic resonance spectroscopic studies on  
some zerovalent platinum phosphine complexes. Al-Ohaly, Abdul-Razzak;  
Nixon, John F. (Sch. Mol. Sci., Univ. Sussex, Brighton/Sussex, BN1 9QJ,  
UK). Inorganica Chimica Acta, 47(1), 105-9 (English) 1981.  
CODEN: ICHAA3. ISSN: 0020-1693.

AB PtLL' [L = 1,1,1-tris(diphenylphosphinomethyl)ethane, L' = PF(CF3)2,  
PPhCl2, PF2(OPh), PCl3, PF2CHCl2] were prepd. by displacement of PPh2Me  
from PtL(PPh2Me). Only the complex PtL(PF2OPh) was sol. enough to obtain  
a satisfactory 31P NMR spectrum exhibiting the expected 1-2-1 triplet of

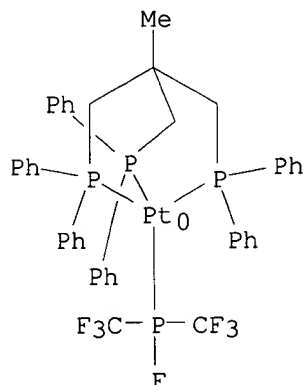
quartets at low field for the PF<sub>2</sub>OPh ligand (coupling to F and P's of L) with <sup>195</sup>Pt satellites and a high field L resonance appearing as a doublet of triplets (coupling to PF<sub>2</sub>OPh) with Pt satellites.

IT **76846-42-9P**

RL: SPN (Synthetic preparation); PREP (Preparation)  
(prepn. of)

RN 76846-42-9 HCA

CN Platinum, [bis(trifluoromethyl)phosphinous fluoride][2-  
[(diphenylphosphino)methyl]-2-methyl-1,3-propanediyl]bis[diphenylphosphine  
]-P,P',P'']-, (T-4)- (9CI) (CA INDEX NAME)



L36 ANSWER 3 OF 13 HCA COPYRIGHT 2003 ACS

72:84687 Phosphorus-fluorine compounds. XVIII. Some N.M.R. observations on tetrakis(fluorophosphine) and mixed fluorophosphine carbonyl complexes of nickel(0). Lynden-Bell, Ruth M.; Nixon, J. F.; Schmutzler, R. (Chem. Lab., Univ. Sussex, Brighton, UK). Journal of the Chemical Society [Section] A: Inorganic, Physical, Theoretical (4), 565-7 (English) 1970. CODEN: JCSIAP. ISSN: 0022-4944.

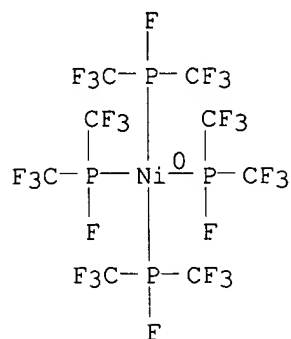
AB Partial analyses of the <sup>19</sup>F NMR spectra of NiL<sub>4</sub> complexes (where L = PF<sub>3</sub>, CF<sub>3</sub>PF<sub>2</sub>, (CF<sub>3</sub>)<sub>2</sub>PF, CCl<sub>3</sub>PF<sub>2</sub>, CH<sub>2</sub>ClPF<sub>2</sub>, Me<sub>2</sub>NPF<sub>2</sub>, and C<sub>6</sub>H<sub>4</sub>O<sub>2</sub>PF) yields values for the coupling consts. 1JPG, 3JPF, 2JPP, and 4JFF. The low values of 2JPP are related to MO energies. NMR parameters for compds. of the type NiLn'-(CO)<sub>4-n</sub> are tabulated [L' = Me<sub>2</sub>NPF<sub>2</sub>, Et<sub>2</sub>NPF<sub>2</sub>, C<sub>5</sub>H<sub>10</sub>NPF<sub>2</sub>, and (Me<sub>2</sub>N)<sub>2</sub>PF].

IT **14917-18-1**

RL: PRP (Properties)  
(nuclear magnetic resonance of)

RN 14917-18-1 HCA

CN Nickel, tetrakis[bis(trifluoromethyl)phosphinous fluoride-P]-, (T-4)-  
(9CI) (CA INDEX NAME)



L36 ANSWER 5 OF 13 HCA COPYRIGHT 2003 ACS

70:120662 Phosphorus-fluorine compounds. XIV. Direct syntheses of tetrakis(fluorophosphine) complexes of zerovalent nickel. Nixon, John F.; Sexton, Michael D. (Univ. Sussex, Brighton, UK). Journal of the Chemical Society [Section] A: Inorganic, Physical, Theoretical (7), 1089-91 (English) 1969. CODEN: JCSIAP. ISSN: 0022-4944.

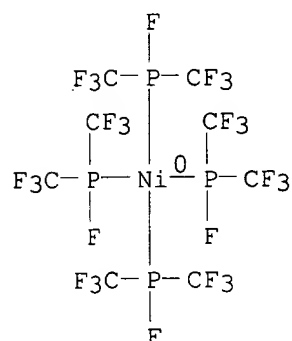
AB Metallic Ni (formed by decarboxylation of Ni oxalate) reacts directly with several fluorophosphines,  $\text{R}_n\text{PF}_3-n$ , at 60.degree. to produce the corresponding zerovalent Ni complex  $\text{NiL}_4$  [ $\text{L} = \text{PF}_3$ ,  $\text{CF}_3\text{PF}_2$ ,  $(\text{CF}_3)_2\text{PF}$ ,  $\text{CCl}_3\text{PF}_2$ ,  $\text{Me}_2\text{NPF}_2$ , and  $\text{C}_5\text{H}_{10}\text{NPF}_2$ ]. The complex  $\text{Ni}[\text{ClCH}_2\text{PF}_2]_4$  which could not be formed in this way was made by the reaction of the ligand with nickelocene; its ir spectrum and F, P, and proton N.M.R. spectra are presented. Anal. of the  $^{19}\text{F}$  N.M.R. spectrum gives an accurate value for  $|1\text{JPF} + 33\text{JPF}'|$  and approx. values for  $|1\text{JPF}|$ ,  $|3\text{JPF}|$ ,  $|2\text{JPP}|$  and  $|4\text{JFF}'|$ .  $^{31}\text{P}$  Chem. shift data for all the complexes are presented and discussed.

IT 14917-18-1P

RL: SPN (Synthetic preparation); PREP (Preparation)  
(prepn. of)

RN 14917-18-1 HCA

CN Nickel, tetrakis[bis(trifluoromethyl)phosphinous fluoride-P]-, (T-4)-  
(9CI) (CA INDEX NAME)



L36 ANSWER 7 OF 13 HCA COPYRIGHT 2003 ACS

68:34531 Chemistry of phosphorus-fluorine compounds. VIII. Synthesis and nuclear magnetic resonance spectra of tetrakisfluorophosphine derivatives of zerovalent nickel. Nixon, John F. (Univ. St. Andrews, St. Andrews, UK). Journal of the Chemical Society [Section] A: Inorganic, Physical, Theoretical (7), 1136-9 (English) 1967. CODEN: JCSIAP. ISSN: 0022-4944.

AB Fluorophosphine complexes of zerovalent Ni of the type  $NiL_4$  [where  $L = PF_3, CF_3PF_2, (CF_3)_2PF, CCl_3PF_2, Et_2NPF_2$ , and  $C_5H_{10}NPF_2$ ] are readily synthesized by the reaction between nickelocene and the fluorophosphine, often under very mild conditions. This synthetic approach avoids difficulties encountered in displacement reactions with Ni tetracarbonyl which invariably lead to  $NiLn(CO)_{4-n}$  mixts. The ir and  $^1H$  and  $^{19}F$  N.M.R. spectra of the complexes are presented and unusual features of the latter are discussed. A significant low-field chem. shift (-17 to -59 ppm.) of the F atoms directly bonded to P, which occurs on complex formation, is attributed to the availability of low-lying excited states in the P-N bonds. 38 references.

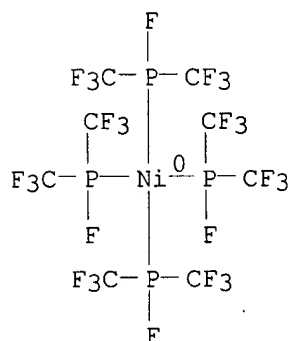
IT **14917-18-1P**

RL: PREP (Preparation)

(prepn. and ir spectrum and N.M.R. of)

RN 14917-18-1 HCA

CN Nickel, tetrakis[bis(trifluoromethyl)phosphinous fluoride-P]-, (T-4)-(9CI) (CA INDEX NAME)



L36 ANSWER 9 OF 13 HCA COPYRIGHT 2003 ACS

65:63118 Original Reference No. 65:11740h,11741a Perfluoromethylphosphine-nickel compounds, including a new volatile heterocycle. Burg, Anton B.; Street, G. Bryan (Univ. of Southern California, Los Angeles). Inorg. Chem., 5(9), 1532-7 (English) 1966.

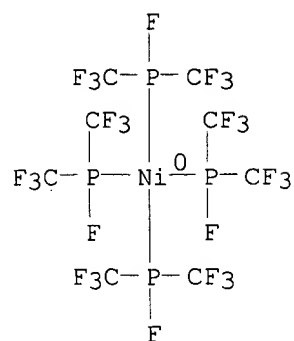
AB The displacement of 3CO from  $Ni(CO)_3$  by  $(CF_3)_2PF$  occurs as readily as the displacement of 2CO by  $CF_3PF_2$ , but this method arrives at pure  $(CF_3PF_2)_2Ni$  (m.p. -84.degree., b.p. estd. 160.degree.) far more easily than pure  $[(CF_3)_2PF]_4Ni$  (air-stable, m.p. 57.8.degree., b.p. estd. 218.degree.), for steric reasons such as prevent 3rd-stage action by  $(CF_3)_3P$ . The ir spectra suggest a slight increase of C-O bond strength as  $CF_3$  replaces F in  $PF_3-Ni-CO$  compds., and the effect is ascribed to minor differences in .pi. bonding. The action of  $PF_3$  upon  $(CF_3PF_4)_2Ni$  relates to the hypothesis that compds. of the type  $Li_2NiL'_2$  often will be more stable than others having different proportions of the same ligands. The new chelate compd.  $(CF_3)_2PC_2F_4P(CF_3)_2Ni(CO)_2$  (m.p. 30.degree., b.p. estd. 179.degree.) is more volatile but less stable than the similar  $C_2H_4$ -connected compd.: complete displacement of the ligand  $(CF_3)_2PC_2F_4P(CF_3)_2$  and 1 CO leads to the product  $[(CF_3)_2PC_2H_4P(CF_3)_2]_2NiCO$  with one bisphosphine unit acting unfunctionally.

IT **14917-18-1**, Nickel, tetrakis[bis(trifluoromethyl)phosphinous fluoride]-

(prepn. of)

RN 14917-18-1 HCA

CN Nickel, tetrakis[bis(trifluoromethyl)phosphinous fluoride-P]-, (T-4)-(9CI) (CA INDEX NAME)



L36 ANSWER 11 OF 13 HCA COPYRIGHT 2003 ACS

64:71694 Original Reference No. 64:13440b-c Kinetics of the decomposition of BH<sub>3</sub>PF<sub>3</sub> and related compounds. A revised estimate of the dissociation energy of diborane. Burg, Anton B.; Chin Fu, Yuan (Univ. of Southern California, Los Angeles). J. Am. Chem. Soc., 88(6), 1147-51 (English) 1966. CODEN: JACSAT. ISSN: 0002-7863.

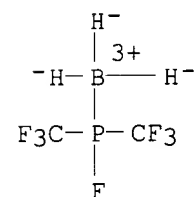
AB Very sensitive ir procedures were used to show that the compds. BH<sub>2</sub>PF<sub>3</sub>, BH<sub>3</sub>.CF<sub>3</sub>PF<sub>2</sub>, and BH<sub>3</sub>.(CH<sub>3</sub>)<sub>2</sub>PF all decomp. to B<sub>2</sub>H<sub>6</sub> and free phosphine ligand by the same mechanism as found earlier for BH<sub>3</sub>CO, namely, the dissocn. of BH<sub>3</sub>L to BH<sub>3</sub> and free ligand L, followed by action of BH<sub>3</sub> to displace L from BH<sub>3</sub>L. The same rate law applies also to the far more complicated case of B<sub>4</sub>H<sub>3</sub>PF<sub>3</sub>. Extrapolation of early-stage rate data for BH<sub>3</sub>PF<sub>3</sub> to zero time gave 1st-order rate consts. for the initial dissocn. at 3 temps. These results, taken with the over-all equil., led to D(BH<sub>3</sub>-BH<sub>3</sub>) = 35.0 kcal., consistent with but more precise than earlier estimates. The only systematic error here would arise from the reasonable assumption that .delta.H = 0 for activation of the reverse of the initial dissocn.

IT 2357-59-7, Borane, compd. with bis(trifluoromethyl)phosphinous fluoride (1:1)

(decompn. of, kinetics of)

RN 2357-59-7 HCA

CN Phosphinous fluoride, bis(trifluoromethyl)-, compd. with borane (1:1) (8CI) (CA INDEX NAME)



L36 ANSWER 13 OF 13 HCA COPYRIGHT 2003 ACS

52:104365 Original Reference No. 52:18461d-i,18462a-d An intramolecular cleavage-cyclization reaction of silicon-containing organolithium compounds. Wittenberg, Dietmar; Gilman, Henry (Iowa State Coll., Ames). J. Am. Chem. Soc., 80, 2677-80 (Unavailable) 1958. CODEN: JACSAT. ISSN: 0002-7863.

AB Br(CH<sub>2</sub>)<sub>4</sub>Br (I) (24.6 g.) in 150 cc. dry Et<sub>2</sub>O added slowly with stirring to 5.0 g. Li sand in 50 cc. Et<sub>2</sub>O, the mixt. stirred a few min. at room temp. to initiate the reaction, then kept at -10 to -20.degree. stirred after the completion of the addn. 1 hr. at -10.degree. and 0.5 hr. at + 10.degree., and filtered through glass wool, the filtrate added slowly

with stirring to 55 g.  $\text{Ph}_3\text{SiCl}$  in 50 cc. dry  $\text{Et}_2\text{O}$ , stirred 1 hr. at  $-10^\circ$ , warmed to room temp., and filtered after 3 hrs., the residue (18 g.) collected, the org. layer of the filtrate washed with  $\text{H}_2\text{O}$  and distd. to remove the volatile materials, the distn. residue chromatographed on  $\text{Al}_2\text{O}_3$ , and the combined original filter residue and eluates distd. gave 9.2 g. 1,1-diphenylsilacyclopentane (II), b<sub>5</sub>  $159-65^\circ$ ,  $n_{\text{D}20}$  1.5855, a 2nd fraction, b<sub>5</sub>  $210-30^\circ$ , which crystd. from MeOH gave 1.3 g.  $\text{Ph}_3\text{SiBu}$ , m.  $85-6^\circ$ , and 16 g. distillate, b<sub>0.05</sub>  $180-200^\circ$ , which crystd. from  $\text{C}_6\text{H}_6$ -EtOH gave 6.5 g.  $\text{Ph}_4\text{Si}$  (III), m.  $232-4^\circ$ ; the distn. residue dissolved in  $\text{C}_6\text{H}_6$ , filtered, and repptd. with ligroine, b.  $60-70^\circ$ , gave 2.5 g.  $(\text{Ph}_3\text{SiCH}_2\text{CH}_2)_2$  (IV), m.  $215-16^\circ$ .  $(\text{CH}_2)_4\text{Li}_2$  from 21.6 g. I added slowly at  $-20^\circ$  with stirring to 25.3 g.  $\text{Ph}_2\text{SiCl}_2$ , stirred 1 hr. at  $-20^\circ$ , warmed to room temp., and hydrolyzed after 1 hr., the org. layer worked up, and the residue chromatographed on  $\text{Al}_2\text{O}_3$  yielded 10.8 g. II, b<sub>5</sub>  $159-62^\circ$ ,  $n_{\text{D}25}$  1.5853, MRD 77.21.  $\text{Si}_2\text{Ph}_6$  (V) cleaved with Li at room temp. in tetrahydrofuran, 35 millimoles of the resulting  $\text{Ph}_3\text{SiLi}$  added with stirring to 3.8 g. I, and filtered, and the residue washed with  $\text{Et}_2\text{O}$  and  $\text{H}_2\text{O}$  yielded 6.65 g. V, m.  $363-6^\circ$ ; the org. layer of the filtrate washed with  $\text{H}_2\text{O}$  and evapd. gave 0.30 g. IV, m.  $215-16^\circ$  (cyclohexane). V (30 g.) in 100 cc. tetrahydrofuran stirred 6 hrs. at room temp. with 5.0 g. Li, filtered through glass wool, heated in a sealed tube slowly to  $125^\circ$ , kept 3 hrs. at  $125^\circ$ , cooled (opened under N), hydrolyzed with dil. acid, and dild. with  $\text{Et}_2\text{O}$ , and the org. layer worked up gave 27.3 g.  $\text{HO}(\text{CH}_2)_4\text{SiPh}_3$  (VI), m.  $110-11^\circ$  (ligroine, b.  $60-70^\circ$ ). VI (12.4 g.) and 5.4 g.  $\text{PBr}_3$  heated 10 hrs. in a steam bath, cooled to room temp., dild. with  $\text{Et}_2\text{O}$ , and hydrolyzed, the org. layer worked up, and the residue dissolved in ligroine and chromatographed on  $\text{Al}_2\text{O}_3$  yielded 8.05 g.  $\text{Br}(\text{CH}_2)_4\text{SiPh}_3$  (VII), m.  $87.5-8.5^\circ$  (MeOH). VII (7.9 g.) and 1.0 g. Li treated with 35 cc. dry  $\text{Et}_2\text{O}$ , cooled to  $-10^\circ$ , stirred until the Li became shiny, cooled to  $-25^\circ$ , stirred at  $-25^\circ$  until the halide had dissolved, stirred 0.5 hr., warmed to room temp., carbonated, hydrolyzed, and extd. with aq. alkali, the combined aq. layers acidified and extd. with  $\text{Et}_2\text{O}$ , and the  $\text{Et}_2\text{O}$  ext. worked up gave 1.37 g.  $\text{BzOH}$ ; the neutral org. layer dried and distd. gave 0.95 g. mixt. of II and  $\text{BzPh}$ , b<sub>18</sub>  $179-91^\circ$ , and 2.4 g. II, b<sub>18</sub>  $191-3^\circ$ ,  $n_{\text{D}20}$  1.5878; the distn. residue recrystd. from  $\text{C}_6\text{H}_6$ -petr. ether gave 0.15 g. III. VII (5.5 g.) and 1.0 g. Li in 40 cc. dry  $\text{Et}_2\text{O}$  allowed to react at  $-25^\circ$ , cooled to  $-70^\circ$ , and treated with Dry Ice, the unreacted Li removed mechanically, the mixt. hydrolyzed, the org. layer extd. with aq. alkali, the combined aq. layers acidified and extd. with  $\text{Et}_2\text{O}$ , the  $\text{Et}_2\text{O}$  ext. evapd., and the crude residue (2.9 g.) extd. with 200 cc. boiling  $\text{H}_2\text{O}$  left 2.8 g.  $\text{HO}_2\text{C}(\text{CH}_2)_4\text{SiPh}_3$  (VIII), m.  $127-9^\circ$ ; the aq. soln. extd. with  $\text{Et}_2\text{O}$  yielded 70 mg.  $\text{BzOH}$ ; the neutral org. layer dried and evapd., and the residue chromatographed from ligroine on  $\text{Al}_2\text{O}_3$  yielded 0.35 g. material consisting mainly of II and 0.05 g. III; further elution with  $\text{C}_6\text{H}_6$  gave 0.12 g.  $[\text{Ph}_3\text{Si}(\text{CH}_2)_4]_2\text{CO}$ , m.  $162-3^\circ$ ; final elution with  $\text{Me}_2\text{CO}$  yielded 0.26 g. VI. VII (5.7 g.) in 30 cc. dry  $\text{Et}_2\text{O}$  added dropwise with stirring to 4.5 g. Mg, refluxed 1 hr. with stirring, carbonated, and hydrolyzed, the org. layer extd. with aq. alkali, the combined acidified aq. solns. extd. with  $\text{Et}_2\text{O}$ , and the ext. worked up gave 3.1 g. VIII, m.  $128-9^\circ$  ( $\text{EtOAc}$ -ligroine); the neutral org. layer distd. and the residue chromatographed on  $\text{Al}_2\text{O}_3$  gave 0.12 g. III and 0.8 g.  $[\text{Ph}_3\text{Si}(\text{CH}_2)_4]_2$ , m.  $132-3^\circ$ .  $\text{Br}(\text{CH}_2)_5\text{Br}$  (23.0 g.) in 150 cc. dry  $\text{Et}_2\text{O}$  added dropwise with stirring to 4.0 g. Li in 50 cc.  $\text{Et}_2\text{O}$ , stirred 1 hr. at  $-10^\circ$  and 0.5 hr. at  $0^\circ$ , filtered through glass wool, and added with stirring to 53 g.  $\text{Ph}_3\text{SiCl}$  in 50 cc.  $\text{Et}_2\text{O}$ , the mixt. stirred 0.5 hr. at  $-10^\circ$ , warmed to room temp., stirred 2 hrs., dild. with  $\text{H}_2\text{O}$ , and filtered, and the residue washed with  $\text{Et}_2\text{O}$  and  $\text{H}_2\text{O}$  and extd. with

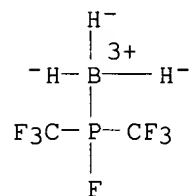
hot EtOH yielded 44.0 g.  $[\text{Ph}_3\text{Si}(\text{CH}_2)_2]_2\text{CH}_2$ , m. 146-6.5.degree.  
(cyclohexane); the combined alc. mother liquor and Et<sub>2</sub>O filtrate evapd.,  
and the residue dissolved in ligroine and chromatographed on Al<sub>2</sub>O<sub>3</sub> gave  
0.25 g. colorless liquid, b<sub>5</sub> 193-8.degree., n<sub>D</sub><sup>20</sup> 1.5779, consisting mainly  
of 1,1-diphenylsilacyclohexane; further elution with ligroine gave 220 mg.  
III, m. 233-5.degree.. The formation of the compds. described is  
explained by an intramol. cleavage-cyclization reaction of the  
intermediate  $\text{Ph}_3\text{Si}(\text{CH}_2)_2\text{aLi}$ .

IT 2357-59-7, Phosphinous fluoride, bis(trifluoromethyl)-, compd.  
with BH<sub>3</sub>

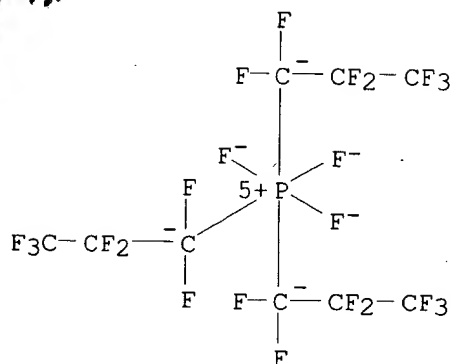
(prepn. of)

RN 2357-59-7 HCA

CN Phosphinous fluoride, bis(trifluoromethyl)-, compd. with borane (1:1)  
(8CI) (CA INDEX NAME)

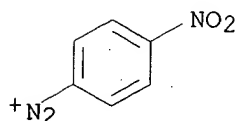






CM 2

CRN 14368-49-1  
CMF C6 H4 N3 O2



IT 123199-70-2P 123199-72-4P 123199-73-5P  
123199-74-6P

RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT  
(Reactant or reagent)  
(prepn. and thermal decompn. of)

L25 ANSWER 2 OF 3 HCA COPYRIGHT 2003 ACS  
ACCESSION NUMBER: 71:81483 HCA

TITLE: Formation of trifluoromethylated fluoro phosphates by  
reaction of trimethyltrifluoromethyltin with  
phosphorus(V) fluoride

AUTHOR(S): Jander, Jochen; Boerner, Dieter; Engelhardt, Udo  
CORPORATE SOURCE: Freie Univ., Berlin, Fed. Rep. Ger.  
SOURCE: Justus Liebig's Annalen der Chemie (1969),  
726, 19-24

CODEN: JLACBF; ISSN: 0075-4617

DOCUMENT TYPE: Journal

LANGUAGE: German

AB PF5 reacted with Me3SnCF3 to give a white hygroscopic ppt. that slowly  
gave off PF5; the anions formed are pptd. from CH2Cl2 as stable  
(Ph4As)(PF5CF3) and (Ph4As)[PF4(CF3)2]. Their structures were established  
from ir and 19F N.M.R. data and a mechanism of formation is discussed.

IT 23940-75-2P

RL: SPN (Synthetic preparation); PREP (Preparation)  
(prepn. of)

RN 23940-75-2 HCA

CN Arsonium, tetraphenyl-, tetrafluorobis(trifluoromethyl)phosphate(1-) (8CI)  
(CA INDEX NAME)

sang-Foster

09/918,464

05/02/2003

100.0% PROCESSED 15392 ITERATIONS  
SEARCH TIME: 00.00.01

98 ANSWERS

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FILE LAST UPDATED: 1 May 2003 (20030501/ED)

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=> d L25 1-3 ibib abs hitstr hitrn

L25 ANSWER 1 OF 3 HCA COPYRIGHT 2003 ACS  
ACCESSION NUMBER: 111:174247 HCA  
TITLE: Reaction of tris(perfluoroalkyl)phosphine oxides and tris(perfluoroalkyl)difluorophosphoranes with fluoride ion  
AUTHOR(S): Pavlenko, N. V.; Yagupol'skii, L. M.  
CORPORATE SOURCE: Inst. Org. Khim., Kiev, USSR  
SOURCE: Zhurnal Obshchei Khimii (1989), 59(3), 528-34  
CODEN: ZOKHA4; ISSN: 0044-460X  
DOCUMENT TYPE: Journal  
LANGUAGE: Russian  
OTHER SOURCE(S): CASREACT 111:174247  
AB Treating (C2F5)3P(O) with 1 or 2 equiv. CsF in Et2O gave (C2F5)3PFOCs or (C2F5)2PF2OCs, resp.; hydrolysis of the latter gave C2F5P(O)FOCs. Treating R3PF2 (R = C2F5, C3F7, C4F9) with MF (M = Cs, K, Na) in Et2O gave quant. M+[R3PF3]-. Diazotization of 4-XC6H4NH2 (X = Cl, Me, NO2) and subsequent reaction with K+[R3PF3]- (R = C2F5, C3F7) gave 77-88% [4-XC6H4N2]+[R3PF3]-.  
IT 123199-70-2P 123199-72-4P 123199-73-5P  
123199-74-6P  
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)  
(prepn. and thermal decompn. of)  
RN 123199-70-2 HCA  
CN Benzenediazonium, 4-chloro-, (OC-6-21)-trifluorotris(pentafluoroethyl)phosphate(1-) (9CI) (CA INDEX NAME)